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Final Report for Solvent Extraction Bench-scale Treatability Study

Rocky Flats Environmental Technology Site

U.S. Department of Energy
Rocky Flats Field Office
Golden, Colorado

Environmental Restoration Program

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1.0 INTRODUCTION

This Final Report has been prepared by Resources Conservation Company (RCC) and their sub-contractor, Harding Lawson Associates, as a contract deliverable between EG&G Rocky Flats Environmental Technology Site (RFETS) (for the Environmental Restoration Program) and RCC. The purpose of this Final Report is to describe the technical approach, results, and assessment of Phase I and Phase II of the solvent extraction bench-scale treatability study program.

1.1 Objectives

The bench-scale treatability study program was performed in two separate phases. Each phase was designed to address feasibility study (FS) data needs regarding the effectiveness of solvent extraction to remove radionuclides from RFETS soil and vegetation. The specific purpose and objectives of Phase I and Phase II of the solvent extraction program are described below. In addition to Phases I and II, a soil particle size and contaminant distribution test was also performed to provide data regarding the distribution of plutonium in RFETS soil as a function of particle size.

1.1.1 Phase I Bench-scale Program Purpose and Objectives

The purpose of Phase I of the bench-scale treatability study program was to provide data to support the FS in assessing the feasibility of using solvent extraction to remediate radionuclide-contaminated RFETS soil and vegetation. To fulfill the purpose of this treatability study, the following objectives were established for Phase I testing: (1) generate performance data for removing contaminants of concern (COCs) from contaminated RFETS soil and vegetation; (2) identify near optimum operating parameters (i.e., number of extraction stages, extraction temperature, pH, solvent ratios, and pretreatment requirements) for removing COCs from contaminated RFETS soil and vegetation using triethylamine; (3) calculate the percent of total plutonium-239 and plutonium-240 (defined as Pu-239,240) removed from RFETS soil and vegetation after solvent extraction testing; (4) calculate Pu-239,240 and solids mass balance for each of the solvent extraction tests performed during Phase I;

and (5) evaluate the potential of Phase II bench-scale testing to remove COCs from the soil and vegetation samples to concentrations at or below the treatability study benchmarks (TSBs) shown in Table 1.1.

1.1.2 Phase II Bench-scale Program Purpose and Objectives

The purpose of Phase II of the bench-scale treatability study program was to provide additional data to support the FS in assessing the feasibility of using solvent extraction to remediate radionuclide-contaminated RFETS soil and vegetation. To fulfill the purpose of this treatability study, the following objectives were established for Phase II testing: (1) confirm the reproducibility of the Pu-239,240 percent removal results obtained during Phase I testing for the most favorable tests; (2) evaluate the effect of additional extraction stages on removal of Pu-239,240 from RFETS soil and vegetation beyond that tested in Phase I; (3) calculate the percent of Pu-239,240 removed from RFETS soil and vegetation after solvent extraction testing; (4) calculate a Pu-239,240 and solids mass balance for each of the solvent extraction tests performed during Phase II; and (5) evaluate the performance of Phase II bench-scale tests in removing COCs from the soil and vegetation samples to concentrations at or below the TSBs identified during Phase I.

1.1.3 Soil Particle Size and Contaminant Distribution Test Purpose and Objectives

The purpose of the soil particle size and contaminant distribution testing was to provide data regarding the distribution of Pu-239,240 in RFETS feed soil as a function of particle size. To fulfill the purpose of this test, the following objectives were established: (1) determine the weight distribution of RFETS soil as a function of particle size; (2) determine the distribution of Pu-239,240 in RFETS soil as a function of particle size; and (3) evaluate the ability of a dispersing agent, sodium hexametaphosphate (SHMP), to transport Pu-239,240 from the soil phase to the liquid phase.

1.2 Report Organization

The remainder of this report presents a description and discussion of the solvent extraction bench-scale testing program. The sample preparation and technical approach for the Phase I and Phase II solvent extraction bench-scale tests, including the soil particle size distribution testing, are described in Section 2.0. Section 3.0 describes the design of the analytical and quality assurance/quality control (QA/QC) program. The bench-scale test, analytical, mass balance, and contaminant percent removal results are presented and discussed for Phase I and Phase II in Sections 4.0 and 5.0, respectively. In addition, Section 5.0 presents and discusses the results of the particle size distribution testing. A QA/QC summary of the analytical results is presented in Section 6.0. Summary and conclusions of both phases for the solvent extraction treatability study testing program are presented in Section 7.0. Appendix A presents supplemental analytical results for Phase I and Phase II testing, and Appendix B presents backup documentation for the Phase I and Phase II mass balance calculations.

Table 1.1: Treatability Study Benchmarks

Radionuclide Parameters	Concentration (pCi/g)
Americium-241*	2.38
Plutonium-239, 240*	3.65
Uranium (Total)*	144

Source: Programmatic Risk-Based Preliminary Remediation Goals, USDOE RFETS, Golden, CO, Final, July 1994.

pCi/g Picocuries per gram

* Programmatic Preliminary Remediation Goal (PRG) - Draft

2.0 BENCH-SCALE TEST DESIGN

This section describes the bench-scale test design for the Phase I and Phase II solvent extraction tests, in addition to the soil particle size and contaminant distribution test performed in Phase II. The solvent extraction bench-scale tests were designed to determine the effectiveness of solvent extraction technology in removing COCs from RFETS soil and vegetation. The soil particle size and contaminant distribution tests were designed to determine the plutonium distribution in RFETS soil as a function of particle size and to evaluate the ability of SHMP to transport plutonium to the liquid phase.

2.1 Phase I Bench-scale Test Design

Phase I bench-scale testing consisted of sample preparation and bench-scale tests. Soil sample preparation involved screening, blending, and splitting two separate soil samples into analytical samples and bench-scale test samples. Vegetation sample preparation involved cutting, blending, and splitting the vegetation sample into analytical samples and bench-scale test samples. The Phase I bench-scale tests were subdivided into two categories: screening tests and solvent extraction tests. Screening tests were performed to identify oxidizing, reducing, and complexing agents or other potential solubilizing agents showing the highest plutonium removal efficiencies for both soil and vegetation samples. The solvent extraction tests were performed using triethylamine and those reagents identified in the screening test (that showed Pu-239,240 removal of 20 percent or greater) to further evaluate the process' ability to remove COCs. The specific technical approaches for sample preparation, screening tests, and solvent extraction tests are described in the following sections.

2.1.1 Phase I Sample Preparation

Soil and vegetation samples selected by EG&G from RFETS were submitted to the laboratory for sample preparation, chemical characterization, and bench-scale testing. The laboratory prepared the soil samples by screening, blending, and splitting each sample into separate test and analytical

samples. The solvent extraction bench-scale contractor prepared the vegetation sample by blending and splitting the vegetation sample into separate test and analytical samples. The sample preparation for soil sample #1, soil sample #2, and the vegetation sample is described in further detail in the following sections.

2.1.1.1 Phase I Sample Preparation - Soil Sample #1

A standard Tyler sieve was used to remove material greater than 1/4 inch in diameter from soil sample #1. The amount of oversize material was recorded and the oversize material was set aside for return to RFETS (only material less than 1/4 inch in diameter was treated and characterized during bench-scale treatability testing). For soil samples #1 and #2, 45 and 27 percent of the soil by weight was less than 1/4 inch, respectively.

The blending process involved splitting the screened sample into two portions, recombining the split sample, and mixing the recombined soil thoroughly. The splitting, recombining, and mixing (homogenization) step was performed a minimum of eight times. Following the homogenization step, soil sample #1 was split into analytical and bench-scale test samples as shown in Figure 2.1. The analytical samples were further divided into chemical characterization samples and submitted to the laboratory for chemical analysis. Chemical analysis of these samples provided baseline chemical characterization data to evaluate the effectiveness of the blending process and plutonium removal efficiencies during bench-scale testing. The prepared bench-scale test samples were stored until required for Phase I and Phase II testing.

2.1.1.2 Phase I Sample Preparation - Soil Sample #2

Preparation of soil sample #2 consisted of screening, blending, and splitting as described in Section 2.1.1.1. Following the screening and blending (homogenization) step, soil sample #2 was split into analytical and bench-scale test samples as shown in Figure 2.1. The analytical sample was

further divided into chemical characterization samples and submitted to the laboratory for chemical analysis. Chemical analysis of these samples provided baseline chemical characterization data to evaluate the effectiveness of the blending process and to evaluate plutonium removal efficiencies during bench-scale testing. The prepared bench-scale test samples were stored until required for Phase I and Phase II testing.

2.1.1.3 Phase I Sample Preparation - Vegetation Sample

The "as received" vegetation sample consisted of two root balls and their accompanying stems and leaves. The stems and leaves were clipped with scissors and set aside. The root balls were rinsed with water to remove most of the soil adhering to the surface of the vegetation. Any floating material was skimmed off the water surface and set aside.

The remaining root balls, stems, leaves, and skimmed material were combined and then ground in a meat grinder. The resulting ground vegetation mixture was blended by hand using the split and recombine technique (homogenization step) described in Section 2.1.1.1. Following the homogenization step, analytical and bench-scale test samples were split as shown in Figure 2.2. The analytical samples were further split and submitted to the laboratory for chemical analysis. Chemical analysis of these samples provided baseline chemical characterization data to evaluate the effectiveness of the blending process and to evaluate plutonium removal efficiencies during bench-scale testing.

2.1.2 Phase I Bench-scale Tests

The bench-scale tests for Phase I were subdivided into two categories: screening tests and solvent extraction tests. The results of the screening tests were used to direct the approach in the solvent extraction tests. Both types of tests are described in further detail in the following sections.

2.1.2.1 Phase I Screening Tests

Screening tests were performed to evaluate plutonium removal efficiency using several combinations of oxidizing, reducing, and complexing reagents as well as other potential solubilizing agents. Each screening test consisted of adding one or more reagents and conducting one extraction stage. Generally, an extraction stage consisted of adding reagent, mixing the sample with the reagent solution, separating liquids from solids (centrifugation), and recycling solids to the extraction vessel. Gross alpha screening was performed on the extract solution after each reagent addition to evaluate plutonium removal for each reagent or combination of reagents tested. Screening tests showing greater than approximately 20 percent plutonium removal were tested further using a maximum of six extraction stages. These subsequent extraction stages are referred to as solvent extraction tests. The specific technical approach used during the screening tests for soil sample #1, soil sample #2, and the vegetation sample are described in the following sections.

2.1.2.1.1 Phase I Screening Tests - Soil Sample #1

Fifteen screening tests were performed on soil sample #1 during Phase I testing. A summary of the test parameters used in the soil sample #1 screening tests is provided in Table 2.1. Generally, 100 grams of soil were used for each test. Reagents were added to the soil, as shown in Table 2.1, resulting in a liquid-to-solid ratio ranging from 1:1 to 100:1 by weight. The liquid and soil mixture was then agitated for 30 to 60 minutes at temperatures ranging from 34 degrees Fahrenheit (°F) to 190°F. A small aliquot of extract solution was removed after each reagent addition for gross alpha screening (as discussed in Section 3.1.2). Screening tests showing greater than 20 percent gross alpha removal were subjected to additional extraction stages (as described in Section 2.1.2.2), up to a maximum of six extraction stages, with modifications to the screening test parameters. Test 1, which was a control test, used five extraction stages even though plutonium removal of less than 20 percent was anticipated.

2.1.2.1.2 Phase I Screening Tests - Soil Sample #2

Eleven screening tests were performed on soil sample #2 during Phase I testing. A summary of the test parameters used in the soil sample #2 screening tests is provided in Table 2.2. The test parameters used were similar to those described in Section 2.1.2.1.1. For example, 100 grams of soil were used per test, reagents listed in Table 2.2 were added to the soil resulting in a liquid-to-solid ratio ranging from 1:1 to 8:1, the liquid and soil mixture was agitated for 30 to 60 minutes at temperatures ranging from 34°F to 190°F, and a small aliquot of extract solution was removed after each reagent addition for gross alpha screening. Screening tests showing greater than 20 percent gross alpha removal were subjected to additional extraction stages (as described in Section 2.1.2.2), with modifications to the screening test parameters.

2.1.2.1.3 Phase I Screening Tests - Vegetation Sample

Eight screening tests were performed on the vegetation sample during Phase I testing. A summary of the test parameters used in the screening tests is provided in Table 2.3. Generally, the test parameters were similar to those described in Sections 2.1.2.1.1 and 2.1.2.1.2. For example, 50 grams of vegetation were used per test, reagents listed in Table 2.3 were added to the vegetation resulting in a liquid-to-solid ratio of 8:1, the liquid and vegetation mixture was agitated for 30 to 90 minutes at temperatures ranging from 34°F to 190°F, and a small aliquot of extract solution was removed after each reagent addition for gross alpha screening. Screening tests showing greater than 20 percent gross alpha removal were subjected to additional extraction stages (as described in Section 2.1.2.2), with modifications to the screening test parameters.

2.1.2.2 Phase I Solvent Extraction Tests

Solvent extraction tests were performed following each soil and vegetation screening test showing greater than 20 percent gross alpha removal. Each solvent extraction test consisted of subsequent extraction stages following the screening test (the screening test being the first extraction stage of the

solvent extraction test). Generally, each extraction stage consisted of adding reagent, mixing the sample with the reagent solution, separating liquids from solids (centrifugation), and recycling solids to the extraction vessel. After completion of all extraction stages, triethylamine was added to the separated reagent solution, contaminants were concentrated, and water and triethylamine were recycled. (Triethylamine was used to remove the water from the contaminant solution, allowing the water to be recycled without evaporation or other separation techniques). A block diagram of the bench-scale test process is presented in Figure 2.3.

2.1.2.2.1 Phase I Solvent Extraction Tests - Soil Sample #1

Four screening tests (Tests 1, 4, 10, and 15) were subjected to subsequent extraction stages, with modifications to the screening test parameters, as shown in Table 2.4. These subsequent extraction stages will be referred to as solvent extraction tests. A step-by-step description of Test 15 is given below to provide further clarification of the extraction sequence used during Phase I sample testing.

A 100-gram portion of soil sample #1 was placed in a 1-liter extraction vessel. A solution of 3 percent hydrogen peroxide, used as an oxidizing agent, was added to the extraction vessel to achieve a liquid-to-solids ratio of 8 to 1 by weight. The solution was agitated for 60 minutes at 150°F. After stopping the agitation, solids settling characteristics were observed and it was concluded that centrifugation would be required. The solids were then separated from the extract by centrifugation. The extract solution was sampled and analyzed using a gross alpha screening technique to obtain an estimate of the extraction efficiency of the peroxide solution.

Citric acid, used as a complexing agent, was then added to the extraction vessel to obtain a 0.1 molar citric acid solution. The mixture was again agitated for 60 minutes at 160°F. The solids were separated from the extract by centrifugation. The extract solution, referred to as interstage extract solution, was analyzed using a gross alpha screening technique to obtain an estimate of the extraction

efficiency of the peroxide/citric acid solution. This extract solution, free of suspended solids, was sampled and later analyzed for isotopic plutonium. The extracted solids, referred to as interstage solids, were sampled and later analyzed for isotopic plutonium.

The above extraction procedure was repeated three more times for Test 15, starting with the addition of hydrogen peroxide to the solids and liquid remaining in the extraction vessel, for a total of four extraction stages. The final treated solids and final extract solution were later analyzed for total uranium and isotopic plutonium. After the final extraction stage, a composite extract solution was formed by combining the extract solution from each extraction stage. Triethylamine was then added to the composite extract solution to concentrate the contaminants to a minimal volume. (Addition of triethylamine forms a two-phase system; a light phase containing triethylamine and water, and a heavy phase containing the contaminants and a small amount of water). The heavy phase was then analyzed for isotopic plutonium.

2.1.2.2.2 Phase I Solvent Extraction Tests - Soil Sample #2

Four solvent extraction tests (Tests A, B, C, and D) were performed with modifications to the screening test parameters, as shown in Table 2.5. The step-by-step procedure used for soil sample #2 was similar to that discussed in Section 2.1.2.2.1 for soil sample #1.

2.1.2.2.3 Phase I Solvent Extraction Tests - Vegetation Sample

Four solvent extraction tests (Tests V-1, V-2, V-3, and V-7) were performed with modifications to the screening test parameters, as shown in Table 2.6. The step-by-step procedure used for the vegetation sample was similar to that discussed in Section 2.1.2.2.1 for soil sample #1.

2.2 Phase II Bench-scale Test Design

Phase II bench-scale testing consisted of two general components: sample preparation and solvent extraction tests. Sample preparation was performed by rehomogenizing bench-scale test samples

prepared prior to Phase I testing and splitting each soil sample into analytical samples and Phase II bench-scale test samples. Vegetation sample preparation involved rehomogenizing the Phase I vegetation sample and splitting it into an analytical sample and a Phase II bench-scale test sample.

The solvent extraction tests were performed using triethylamine and those reagents and operating conditions identified in Phase I testing that yielded the most favorable plutonium removal results. The specific technical approach of the sample preparation and solvent extraction tests is described in the following sections.

2.2.1 Phase II Sample Preparation

Prior to Phase II, test samples set aside after the Phase II feed preparation step were blended and sampled again prior to commencing Phase II testing to provide additional data regarding sample homogeneity. Additional Phase II sample preparation for soil sample #1, soil sample #2, and the vegetation sample is described in further detail in the following sections.

2.2.1.1 Phase II Sample Preparation - Soil Sample #1

One of the six test samples from Phase I was selected for Phase II testing. The Phase II test sample was split, blended, and recombined eight more times. After the Phase II sample preparation step was completed two aliquots were withdrawn for Phase II testing and one aliquot was withdrawn for chemical analyses, as shown in Figure 2.4.

2.2.1.2 Phase II Sample Preparation - Soil Sample #2

Soil sample #2 was prepared and aliquots withdrawn using the same method described for soil sample #1, and described in Section 2.2.1.1.

2.2.1.3 Phase II Sample Preparation - Vegetation Sample

One of the six test samples from Phase I was selected for Phase II testing. The Phase II test sample was split, blended, and recombined eight more times. After the Phase II sample preparation step was completed two aliquots were withdrawn for Phase II testing and one aliquot was withdrawn for chemical analyses as shown in Figure 2.4.

2.2.2 Phase II Solvent Extraction Tests

Phase II solvent extraction tests were performed using the most effective combination of oxidizing/ reducing agents, complexing agents, triethylamine, extraction time, and extraction temperature identified during Phase I testing. The process operating parameters used in Phase II testing are presented in Table 2.7 for soil sample #1, Table 2.8 for soil sample #2, and Table 2.9 for the vegetation sample. The sample size and number of extraction stages were increased during Phase II testing as compared to Phase I testing (sample sizes were doubled during Phase II testing and 12 extraction stages were conducted during Phase II instead of the 3 or 4 stages used during Phase I testing). Generally, each extraction stage consisted of adding a reagent, mixing the sample with the reagent solution, separating liquids from solids (centrifugation), and recycling solids to the extraction vessel. After completion of all the extraction stages, triethylamine was added to the separated reagent solution, contaminants were concentrated, and water and triethylamine were recovered. (Triethylamine was used to remove the water from the contaminant solution, allowing the water to be recycled without evaporation or other separation techniques). A block diagram of the solvent extraction bench-scale test process is presented in Figure 2.3. Solvent extraction testing procedures used during Phase II for soil sample #1, soil sample #2, and the vegetation sample are described in further detail in the following sections.

2.2.2.1 Phase II Solvent Extraction Tests - Soil Sample #1

Two solvent extraction tests were conducted during Phase II testing of soil sample #1. The test parameters used for each test are presented Table 2.7. A step-by-step description of one of the two tests is given below to provide further clarification of the extraction sequence used during Phase II sample testing.

A 200-gram portion of soil sample #1 was placed in a 1-liter extraction vessel. A solution of 3 percent hydrogen peroxide, used as an oxidizing agent, was added to the extraction vessel to achieve a liquid-to-solids ratio of 8 to 1 by weight. The solution was agitated and heated to 190°F. Citric acid, used as a complexing agent, was then added to the extraction vessel to obtain a 0.1 molar citric acid solution. The mixture was agitated for 60 minutes at 190°F. After stopping the agitation, solids settling characteristics were observed and it was concluded that centrifugation would be required. The solids were then separated from the extract solution by centrifugation. This extract solution, free of suspended solids, was analyzed for isotopic plutonium, americium-241 (Am-241), and total uranium.

The above extraction procedure was repeated 11 more times, starting with the addition of hydrogen peroxide to the solids and liquid remaining in the extraction vessel, for a total of 12 extraction stages. The final treated solids were analyzed for total uranium, isotopic plutonium, and Am-241. After the final extraction stage, two composite extract solutions were formed by combining the extract solutions from extraction stages 1 through 6 and the extract solutions from extraction stages 7 through 12. An aliquot of each of these two composite samples was analyzed for isotopic plutonium, Am-241, and total uranium. These two composite extracts were then combined to form a single extract solution composite. Triethylamine was then added to the extract solution composite to concentrate the contaminants to a minimal volume. (Addition of triethylamine forms a two-phase

system; a light phase containing triethylamine and water, and a heavy phase containing the contaminants and a small amount of water.) The heavy phase, produced by adding triethylamine to the composite extract solution, was then analyzed for isotopic plutonium, Am-241, and total uranium. The water recovered from the extract solution was sampled and later analyzed for isotopic plutonium, Am-241, and total uranium.

2.2.2.2 Phase II Solvent Extraction Tests - Soil Sample #2

Two solvent extraction tests were performed on soil sample #2 using the test parameters shown in Table 2.8. The step-by-step procedure used for soil sample #2 was similar to that discussed in Section 2.2.2.1 for soil sample #1.

2.2.2.3 Phase II Solvent Extraction Tests - Vegetation Sample

One solvent extraction test was performed on the vegetation sample using the test parameters shown in Table 2.9. The step-by-step procedure used for the vegetation sample was similar to that discussed in Section 2.2.2.1 for soil sample #1.

2.3 Soil Particle Size and Contaminant Distribution Testing

A soil particle size and contaminant distribution test was conducted on soil sample #1 and soil sample #2 to determine the weight distribution and isotopic plutonium activity of the feed material as a function of particle size. The test was conducted using 150 grams of feed soil, prepared as described in Section 2.1.1. The feed soil was mixed with 150 milliliters of 0.1 molar SHMP, used as a dispersing agent for the soil slurry. The soil slurry was then poured through a series of three standard screens (#5, #8, and #40 mesh), arranged to capture sequentially smaller particle size fractions. Samples of these fractions were submitted for isotopic plutonium analysis. The material that passed through all three screens was flocculated using Superfloc® 208 to assist in the separation of solids from the liquid. The liquid above the flocculated soil was decanted and filtered using #40 and #41 Whatman filter paper and dried in a forced draft oven. The flocculated solids were dried

and combined with the material recovered via filtration and submitted to the laboratory for isotopic plutonium analysis. The decanted liquid was collected after filtration and submitted for isotopic plutonium analysis.

Table 2.1: Summary of Phase I Screening Test Parameters for Soil Sample #1

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature	Further Testing
1	---	---	Triethylamine	1	60 min	<40°F	Y
2	---	Na_2CO_3	Triethylamine	1	30 min	Room temp	N
3	H_2O_2	Na_2CO_3	Triethylamine	1	30 min	<40°F	N
4	H_2O_2	$C_6H_8O_7$	Triethylamine	1	30 min	150°F *	Y
5	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	1	30 min	<40°F	N
6	HNO_3	$C_6H_8O_7$	Triethylamine	1	30 min	150°F *	N
7	HNO_3	---	---	1	30 min	Room temp	N
8	H_2O_2	$Na_2CO_3/Na_3C_6H_5O_7$	---	1	60 min	160°F	N
9	H_2O_2	---	Aliquot 336™	1	30 min	Room temp	N
10	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	1	60 min	150°F	Y
11	$Na_2S_2O_4$	$C_{12}H_{27}O_4P$	Triethylamine	1	60 min	Room temp	N
12	---	$C_{12}H_{27}O_4P$	Triethylamine	1	30 min	<40°F	N
13	H_2O_2	$C_{12}H_{27}O_4P$	---	1	30 min	160°F	N
14	H_2O_2	$C_6H_8O_7/C_{12}H_{27}O_4P$	---	1	30 min	160°F	N
15	H_2O_2	$C_6H_8O_7$	---	1	60 min	160°F	Y

---	None
<	Less than
$C_6H_8O_7$	Citric acid
$C_{12}H_{27}O_4P$	Tributyl phosphate
HNO_3	Nitric acid
H_2O_2	Hydrogen peroxide
min	Minute
N	No
Na_2CO_3	Sodium carbonate
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
temp	Temperature
Y	Yes
°F	Degrees Fahrenheit

* Hot aqueous extractions followed by cold (<40 °F) triethylamine extractions.

Table 2.2: Summary of Phase I Screening Test Parameters for Soil Sample #2

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature	Further Testing
A	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	1	60 min	160°F	Y
B	H_2O_2	$C_6H_8O_7$	---	1	60 min	190°F	Y
C	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	1	60 min	190°F	Y
D	H_2O_2	$C_6H_8O_7$	Triethylamine	1	30 min	190°F	Y
E	HNO_3	---	Triethylamine Aliquot 336™	1	60 min	190°F	N
F	HNO_3	$C_{12}H_{27}O_4P$	Triethylamine	1	60 min	190°F	N
G	HNO_3	$C_{12}H_{27}O_4P$	Aliquot 336™	1	60 min	Room temp	N
H	HNO_3	---	Sodium Nitrite Aliquot 336™	1	60 min	Room temp	N
J	$Na_2S_2O_4$	---	Hydrochloric Acid, Sodium Chloride	1	60 min	190°F	N
K	H_2SO_4	---	Sodium	1	60 min	190°F	N
L	---	---	Triethylamine	1	30 min	<40°F	N

---	None
$C_6H_8O_7$	Citric acid
$C_{12}H_{27}O_4P$	Tributyl phosphate
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulfuric acid
HNO_3	Nitric acid
min	Minute
N	No
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
temp	Temperature
Y	Yes
°F	Degrees Fahrenheit

Table 2.3: Summary of Phase I Screening Test Parameters for the Vegetation Sample

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature	Further Testing
V-1	H_2O_2	$C_6H_8O_7$	Triethylamine	1	60 min	190°F	Y
V-2	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	1	60 min	190°F	Y
V-3	H_2O_2	$C_6H_8O_7$	Triethylamine	1	60 min	<40°F	Y
V-4	H_2O_2	$Ca(NO_3)_2$	---	1	60 min	Room temp	N
V-5	---	$Ca(NO_3)_2$	---	1	90 min	Room temp	N
V-6	---	$Ca(NO_3)_2$	---	1	90 min	Room temp	N
V-7	NaOCl	---	---	1	30 min	170°F	Y
V-8	HNO_3	$C_6H_8O_6$	---	1	30 min	Room temp	N

---	None
<	Less than
$C_6H_8O_6$	Ascorbic acid
$C_6H_8O_7$	Citric acid
$Ca(NO_3)_2$	Calcium nitrate
H_2O_2	Hydrogen peroxide
HNO_3	Nitric acid
min	Minutes
N	No
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
NaOCl	Sodium hypochlorite
temp	Temperature
Y	Yes
°F	Degrees Fahrenheit

Table 2.4: Summary of Phase I Solvent Extraction Test Parameters for Soil Sample #1

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature
1	---	---	Triethylamine	5	60 min	Ext. #1,2 - Ext. #3-5 -
4	H_2O_2	$C_6H_8O_7$	Triethylamine	3	30 min	150°F *
10	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	4	60 min	150°F
15	H_2O_2	$C_6H_8O_7$	---	4	60 min	160°F

---	None
<	Less than
$C_6H_8O_7$	Citric acid
H_2O_2	Hydrogen peroxide
min	Minutes
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
°F	Degrees Fahrenheit

* Hot aqueous extraction followed by cold (<40°F) triethylamine extraction.

Table 2.5: Summary of Phase I Solvent Extraction Test Parameters for Soil Sample #2

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature
A	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	4	60 min	160°F
B	H_2O_2	$C_6H_8O_7$	---	4	60 min	190°F
C	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	4	60 min	190°F
D	H_2O_2	$C_6H_8O_7$	Triethylamine	4	30 min	190°F

---	None
$C_6H_8O_7$	Citric acid
H_2O_2	Hydrogen peroxide
min	Minutes
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
°F	Degrees Fahrenheit

Table 2.6: Summary of Phase I Solvent Extraction Test Parameters for the Vegetation Sample

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature
V-1	H_2O_2	$C_6H_8O_7$	Triethylamine	6	60 min	190°F
V-2	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	6	60 min	190°F
V-3	H_2O_2	$C_6H_8O_7$	Triethylamine	3	60 min	Ext.#1,2 - <40°F Ext.#3 - 190°F
V-7	NaOCl	---	---	2	30 min	170°F

---	None
<	Less than
$C_6H_8O_7$	Citric acid
H_2O_2	Hydrogen peroxide
min	Minutes
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
NaOCl	Sodium hypochlorite
°F	Degrees Fahrenheit

Table 2.7: Summary of Phase II Solvent Extraction Test Parameters for Soil Sample #1

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature
1	H_2O_2	$C_6H_8O_7$	Triethylamine	12	60 min	190°F
2	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	12	60 min	190°F

$C_6H_8O_7$	Citric acid
H_2O_2	Hydrogen peroxide
min	Minutes
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
°F	Degrees Fahrenheit

Table 2.8: Summary of Phase II Solvent Extraction Test Parameters for Soil Sample #2

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature
1	H_2O_2	$C_6H_8O_7$	Triethylamine	12	60 min	190°F
2	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	12	60 min	190°F

$C_6H_8O_7$	Citric acid
H_2O_2	Hydrogen peroxide
min	Minutes
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
°F	Degrees Fahrenheit

Table 2.9: Summary of Phase II Solvent Extraction Test Parameters for the Vegetation Sample

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time per Stage	Extraction Temperature
1	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	12	60 min	190°F

min	Minutes
$Na_2S_2O_4$	Sodium dithionite
$Na_3C_6H_5O_7$	Sodium citrate
°F	Degrees Fahrenheit

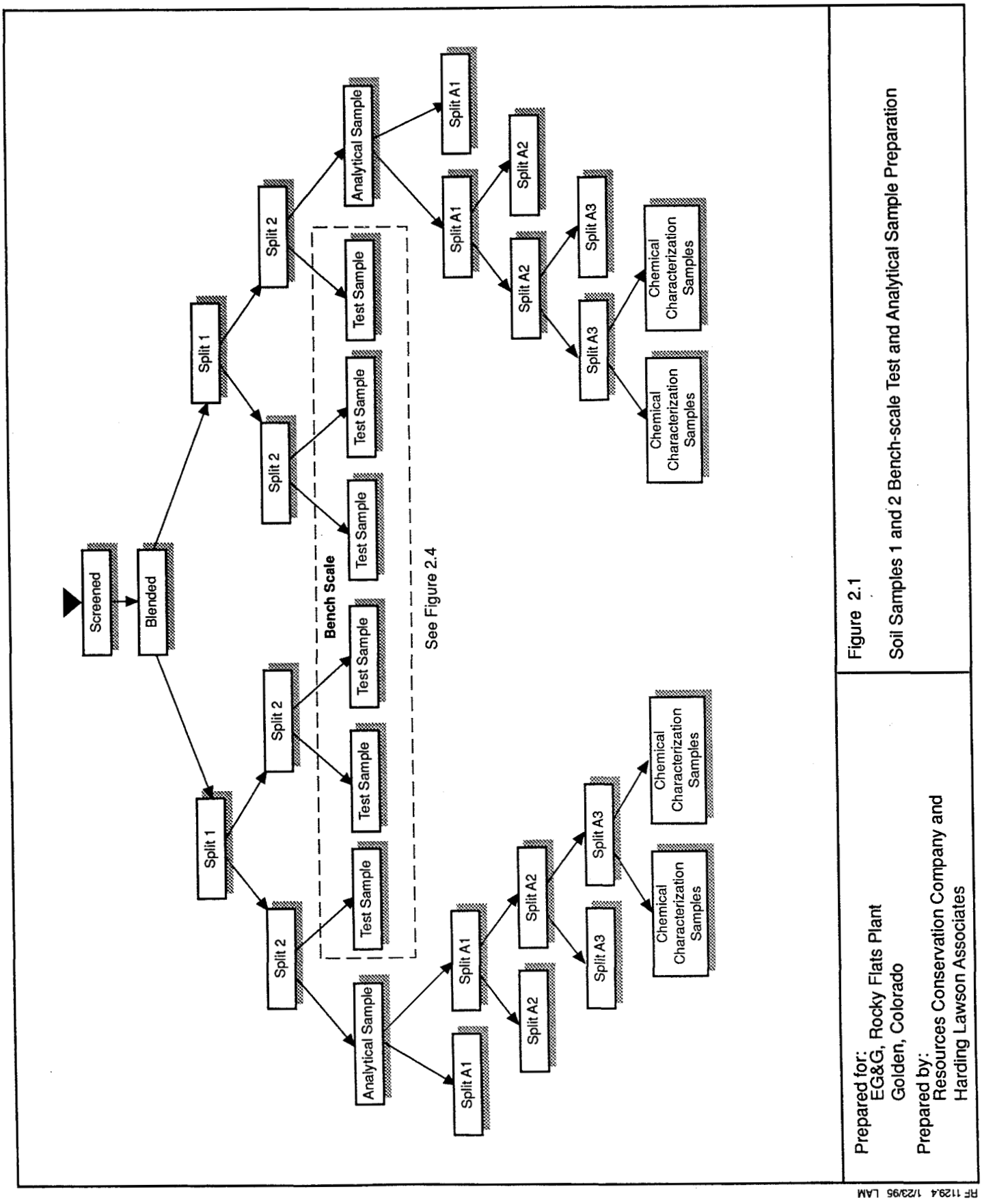

Figure 2.1

Soil Samples 1 and 2 Bench-scale Test and Analytical Sample Preparation

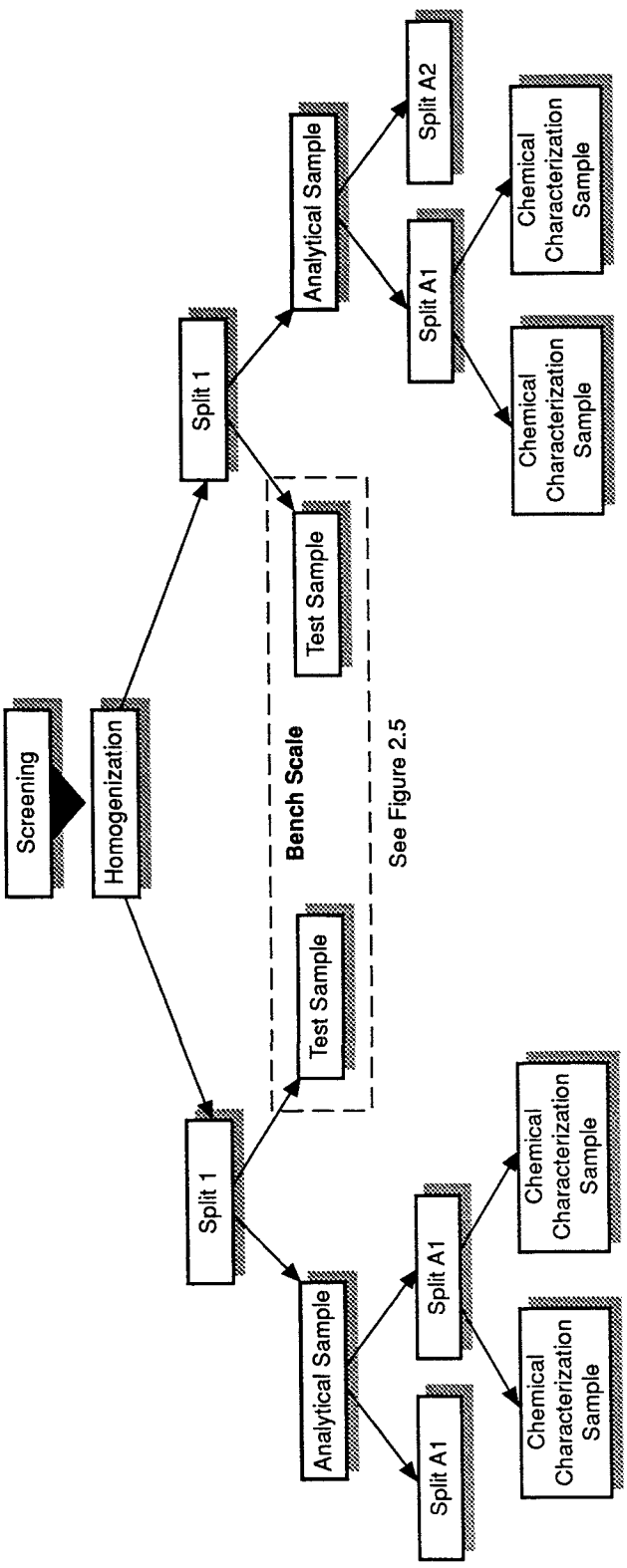

Figure 2.2

Vegetation Bench-scale Test and Analytical Sample Preparation

Prepared for:
EG&G, Rocky Flats Plant
Golden, Colorado

Prepared by:
Resources Conservation Company and
Harding Lawson Associates

RF 1129.3 1/23/95 LAM

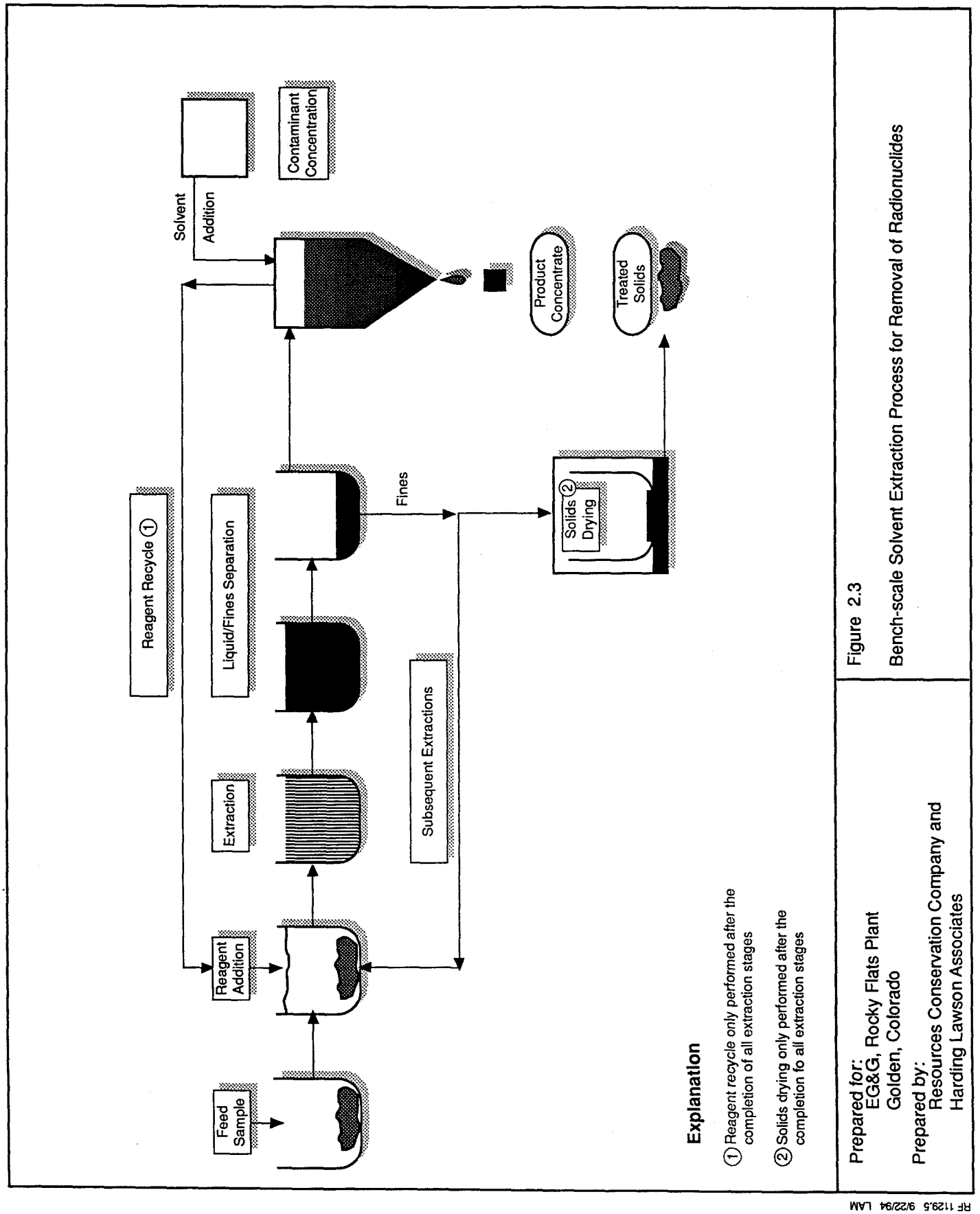

Figure 2.3

Bench-scale Solvent Extraction Process for Removal of Radionuclides

RF 1129.5 9/22/94 LAM

Figure 2.4

Phase II Bench-scale and Analytical Sample Preparation

Prepared for:
EG&G, Rocky Flats Plant
Golden, Colorado

Prepared by:
Resources Conservation Company and
Harding Lawson Associates

RF 1129.6 1/24/95 LAM

3.0 ANALYTICAL AND QUALITY ASSURANCE/QUALITY CONTROL PROGRAM DESIGN

A detailed sampling and analytical program was conducted in support of the solvent extraction bench-scale treatability study to assess the ability of solvent extraction to remove COCs from contaminated RFETS soil and vegetation. This analytical program included quality assurance/quality control (QA/QC) procedures to establish the defensibility of the analytical results. The analytical and QA/QC procedures used for the solvent extraction bench-scale treatability study are summarized below.

3.1 Phase I Analytical Program Design

As described in Section 2.1, the analytical program for the Phase I bench-scale testing included three general components: (1) chemical characterization of the RFETS feed soil and vegetation samples prior to testing, (2) radioactivity screening analyses in support of Phase I bench-scale screening tests, and (3) radiochemical analyses for COCs in support of Phase I bench-scale solvent extraction tests. Sample analyses for all three components of the Phase I analytical program were performed at the laboratory.

3.1.1 Feed Sample Chemical Characterization

Prior to conducting the Phase I bench-scale tests, the feed soil and vegetation samples obtained from RFETS were screened, thoroughly blended, and split into analytical and test samples, as described in Section 2.1. The amount of oversize material was recorded and set aside for return to RFETS. (Only material less than 1/4 inch in diameter was used in bench-scale testing. No testing or characterization of material greater than 1/4 inch in diameter was performed.) The analytical sample splits were submitted to the laboratory to be analyzed for the following radiochemical parameters: Pu-238, Pu-239,240, total uranium (U), americium (Am-241), gross alpha, and gross beta. A portion of several of the bench-scale test samples was also analyzed for Pu-239,240 to further characterize the feed samples. Plutonium and americium isotopes were analyzed using alpha spectrometry, uranium was

analyzed using kinetic phosphorimetry, and gross alpha and gross beta were analyzed using gas proportional counting. The specific analytical protocols applied for the characterization of feed samples followed the laboratory standard operating procedures (SOPs) for radiochemical analyses as documented in the Quality Assurance Addendum (QAA) for the solvent extraction bench-scale treatability study. Results from the feed sample chemical characterization analyses established feed (influent) concentrations of COCs and were used to assess COC removal achieved by the bench-scale tests.

In addition to radiochemical analyses, the analytical sample splits from the feed samples underwent screening analyses for total solids, oil and grease, and pH. These analyses were performed by bench-test technicians using internal SOPs. Results from these analyses assisted in the design of the Phase I bench-scale tests.

3.1.2 Radioactivity Screening Analyses for Phase I Bench-scale Screening Tests

As described in Section 2.1.2, the Phase I bench-scale testing program included screening tests using specific extraction formulations for evaluating the effectiveness of removing COCs from the RFETS feed soil and vegetation samples. These screening tests generally involved performing a single extraction stage. To estimate the effectiveness of the screening test in removing COCs, the extract solution generated from each screening test was submitted to the laboratory to undergo semi-quantitative screening analyses for gross alpha activity. Performance of these screening analyses involved modifying the laboratory's SOP for gross alpha analysis to use a smaller sample aliquot (approximately 1 milliliter), a simplified sample preparation procedure (i.e., direct loading of sample onto a planchette), and an abbreviated counting time. Although the screening analyses produced data with greater associated uncertainty than the laboratory's standard method for gross alpha

analysis, the screening analyses data allowed specific extraction approaches to be evaluated on an expedited basis during Phase I.

3.1.3 Radiochemical Analyses for Phase I Bench-scale Solvent Extraction Tests

Extraction formulations and approaches showing promise based on screening results were more thoroughly investigated by performing additional extraction stages. Process samples from these tests were submitted to the laboratory to be analyzed for specific radiochemical parameters. Process sample types and analytical parameters for these Phase I solvent extraction tests are summarized in Table 3.1. These analyses used the same analytical methods used in the feed sample chemical characterization analyses, and followed the laboratory's SOPs for radiochemical analyses as documented in the QAA for the solvent extraction bench-scale treatability study.

3.2 Phase II Analytical Program Design

As described in Section 2.2, the analytical program for the Phase II bench-scale testing included two general components: (1) chemical analyses of the RFETS feed soil and vegetation samples and (2) radiochemical analyses for COCs in support of Phase II bench-scale solvent extraction tests. Sample analyses for both components of the Phase II analytical program were performed at the laboratory.

3.2.1 Feed Sample Analyses for Phase II Testing

Prior to conducting Phase I testing, soil sample #1 was screened, blended, and split into six separate test samples and two analytical samples, as described in Section 2.1.1.1. One of the six test samples was selected for Phase II testing. The Phase II test sample was split, blended, and recombined an additional eight times. Samples for feed characterization analyses were withdrawn from the homogenized Phase II test sample and from the remaining Phase I feed samples.

The Phase II analytical sample split was submitted to the laboratory to be analyzed for the following radiochemical parameters: Pu-238; Pu-239,240; U-234; U-235; U-238; total uranium; and Am-241. A portion of several of the bench-scale test samples was also analyzed for Pu-239,240 to further characterize the feed samples. Plutonium, uranium, and americium isotopes were analyzed using alpha spectrometry, and total uranium was analyzed using kinetic phosphorimetry. The specific analytical protocols applied for the characterization of feed samples followed the laboratory's SOPs for radiochemical analyses as documented in the QAA for the solvent extraction bench-scale treatability study. Results of the feed sample analyses established the reproducibility of radionuclide analyses for Phase II testing.

In addition to radiochemical analyses, the analytical sample splits from the Phase II feed samples underwent screening analyses for total solids and Toxicity Characteristic Leaching Procedure (TCLP) metals (TCLP metals analyses were not conducted on the vegetation sample). Total solids analyses were performed by bench-test technicians using internal SOPs, and TCLP metals analyses were performed by the laboratory using inductively coupled plasma spectrometry.

3.2.2 Radiochemical Analyses for Phase II Bench-scale Solvent Extraction Tests

Extraction formulations and approaches that showed the greatest promise during Phase I testing, based on percent plutonium removal results, were used during Phase II testing. Additional extraction stages were performed to evaluate the effect of additional extraction stages on COC removal. Process samples from these tests were submitted to the laboratory to be analyzed for specific radiochemical parameters. Process sample types and analytical parameters for these Phase II solvent extraction tests are summarized in Table 3.2. These analyses used the same analytical methods used in the feed sample chemical characterization analyses, and followed the laboratory's SOPs for radiochemical analyses as documented in the QAA for the solvent extraction bench-scale treatability study.

In addition to radiochemical analyses, treated solid samples from soil sample #1 and soil sample #2 were analyzed for oil and grease and TCLP metals.

3.3 Soil Particle Size and Contaminant Distribution Testing Analytical Program Design

A soil particle size and contaminant distribution test was conducted on soil sample #1 and soil sample #2 to assess the weight distribution and radionuclide activity of the feed material as a function of particle size (see Section 2.3). Samples of particle size fractions, flocculated solids, and filtrates from these tests were submitted for Pu-238 and Pu-239,240 analyses according to the laboratory SOPs documented in the QAA.

3.4 Quality Assurance/Quality Control Program

QA/QC protocols were applied during the solvent extraction bench-scale treatability study to establish the quality of the reported analytical results. Multiple internal QC checks were performed during test sample analysis, including the analysis of duplicate test samples, laboratory method blanks, and laboratory control samples. Radiochemical tracers were used as required by method SOPs to monitor the recovery of COCs from test sample matrices. Following sample analysis and results reporting, a data review program was implemented to assess data quality and defensibility, and to identify potential limitations on data useability relative to the objectives of the treatability study.

Details of the QA/QC program for the solvent extraction bench-scale treatability study are provided in the QAA. Data quality issues identified from the application of QA/QC protocols during Phase I and II testing, as they relate to specific test results, are discussed in Sections 4.1 and 5.1, respectively. Overall data quality for the solvent extraction bench-scale treatability study is described in Section 6.0.

Table 3.1: Summary of Phase I Sampling and Analytical Program

Process Sample Type[a]	When Collected	Number of Samples Collected per Test	Analytical Parameters		
			Pu-239,240	Pu-238	Total U
Interstage solids	After each intermediate extraction stage	2 to 4[b]	x	x	
Interstage extract solution	After each intermediate extraction stage	2 to 4[b]	x	x	
Final treated solids	After final extraction stage	1	x	x	x
Final extract solution	After final extraction stage	1	x	x	x
Final extract concentrate (heavy phase)	After combining and concentrating all extract solutions from test	0 to 1[c]	x	x	x

Pu Plutonium
U Uranium

a. Includes major process media sampled during Phase I tests.
b. The total number of extractions performed varied between the different bench-scale tests (see Section 2.1.2.2).
c. Not collected during all Phase I bench-scale tests.

Table 3.2: Summary of Phase II Sampling and Analytical Program

Process Sample Type*	When Collected	Number of Samples Collected per Test	Analytical Parameters			
			Pu-239,240	Pu-238	Am-241	Total U
Interstage solids	After the 6th and 9th extraction stage	2	x	x	x	x
Interstage extract solution	Composite sample of the extract solution from extraction stages 1 through 6 and 7 through 12	2	x	x	x	x
Final treated solids	After final extraction stage	1	x	x	x	x
Final extract concentrate (heavy phase)	After combining and concentrating all extract solutions from test	1	x	x	x	x
Recovered water	After combining and concentrating all extract solutions from test	1	x	x	x	x

Am Americium
Pu Plutonium
U Uranium

* Includes major process media sampled during Phase II tests

4.0 PHASE I TESTING RESULTS AND DISCUSSION

This section presents the results of the Phase I bench-scale testing, as described in Section 2.1, including results for bench-scale solvent extraction tests, sample preparation, evaluation of analytical results, percent contaminant removal calculations, and mass balance calculations.

4.1 Phase I Bench-scale Testing

The Phase I bench-scale tests produced both process and analytical results. Process test results for each soil and vegetation sample included approximate values for extraction temperature, extraction time, solids settling and centrifugation characteristics, oxidation/reduction agent addition, complexing agent addition, feed to reagent (i.e., oxidation/reduction and complexing agent) ratios, and solvent to reagent ratios. Analytical results identified the process parameters achieving the highest percent of plutonium removed for each soil and vegetation sample tested. These analytical and process parameter results from the Phase I bench-scale testing were used to identify the process parameters to be used during Phase II testing. In addition, the analytical results provided data for mass balance calculations. Results from the three components of the bench-scale testing (sample preparation, screening tests, and solvent extraction tests) are summarized in the following sections for soil sample #1, soil sample #2, and the vegetation sample.

4.1.1 Phase I Feed Sample Preparation Results

Following the feed sample screening, homogenization, and splitting described in Section 2.1.1, the chemical characterization analytical samples were submitted to the laboratory for chemical analysis to provide data to evaluate the effectiveness of the blending process. The results of the plutonium characterization analyses for each sample are presented in Table 4.1, which presents the analytical results for both the Phase I and Phase II feed samples. The variations in the vegetation and soil feed concentrations may be attributed to analytical variance and the inherent heterogeneity of the sample matrix (see Section 6.0). A statistical evaluation of the chemical characterization analytical results

was performed and is discussed in Section 4.2.1. Additional analytical results (i.e., uranium, americium, and oil and grease, etc.) for chemical characterization analyses for each sample are presented in Appendix A, Analytical Results.

4.1.2 Soil Sample #1 Test Results

The process data collected during Phase I bench-scale testing of soil sample #1 are summarized as follows:

- The extraction temperatures varied from 34°F to 190°F.
- The extraction times varied from 30 minutes to approximately 14 hours.
- Solids settling times of up to 30 minutes were tested and centrifugation was required.
- The oxidation/reduction and complexing agents tested are presented in Table 2.4.
- The ratio of feed to reagent (i.e., oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, varied from 1:1 to 1:100.
- The ratio of solvent to reagent, expressed on a weight-to-weight basis, varied from pure solvent to a ratio 10:1.

The results of Pu-239,240 and total uranium analyses conducted on soil sample #1 feed, interstage (i.e., first extraction, second extraction, etc.), and final treated solids from Phase I solvent extraction tests are presented in Table 4.2. The analytical results show that in Tests 10 and 15, Pu-239,240 was reduced from a mean feed concentration of 740 pCi/g to 86 pCi/g and 95 pCi/g, respectively, in the final treated solids. Additional analytical results (i.e., extract solution, extract concentrate) from Phase I, soil sample #1 solvent extraction tests are presented in Appendix A, Analytical Results.

4.1.3 Soil Sample #2 Test Results

The process data collected during Phase I bench-scale testing of soil sample #2 are summarized as follows:

- The extraction temperatures varied from 34°F to 190°F.
- The extraction times varied from 30 to 60 minutes.
- Solids settling times of up to 30 minutes were tested and centrifugation was required.
- The oxidation/reduction and complexing agents tested are presented in Table 2.5.
- The ratio of feed to reagent (i.e., oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, was varied from 1:1 to 1:8.
- The ratio of solvent to reagent, expressed on a weight-to-weight basis, was varied from pure solvent to a ratio of 19:1.

The results of Pu-239,240 and total uranium analyses conducted on soil sample #2 feed, interstage (i.e., first extraction, second extraction, etc.), and final treated solids from Phase I solvent extraction tests are presented in Table 4.3. The analytical results show that in Tests A and C, Pu-239,240 was reduced from a mean feed concentration of 1200 pCi/g to 170 pCi/g and 180 pCi/g, respectively, in the final treated solids. Additional analytical results (i.e., extraction solution, extraction concentrate) from Phase I, soil sample #2 solvent extraction tests are presented in Appendix A, Analytical Results.

4.1.4 Vegetation Sample Test Results

The process data collected during Phase I bench-scale testing of the RFETS vegetation sample are summarized as follows:

- The extraction temperatures varied from 34°F to 190°F.
- The extraction times varied from 30 to 90 minutes.
- Solids settling times of up to 30 minutes were tested and centrifugation was required.
- The oxidation/reduction and complexing agents tested are presented in Table 2.6.
- The ratio of feed to reagent (i.e., oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, was 1:8.
- The ratio of solvent to reagent, expressed on a weight-to-weight basis, was 5:1.

The results of Pu-239,240 and total uranium analyses conducted on feed, interstage (i.e., first extraction, second extraction, etc.), and final treated solids from Phase I solvent extraction vegetation testing are presented in Table 4.4. The analytical results show that in Test V-2, Pu-239,240 was reduced from a mean feed concentration of 640 pCi/g to 87 pCi/g in the final treated solids. Because the vegetation samples could not be ground to a small uniform particle size, the observed variability in the interstage solid results may reflect heterogeneities between the small sample aliquots used. Additional analytical results (i.e., extraction solution, extraction concentrate) from Phase I, vegetation sample solvent extraction tests are presented in Appendix A, Analytical Results.

4.2 Evaluation of Phase I Analytical Results

Data evaluation of Phase I analytical results was performed to assist in assessing the overall bench-scale test performance during Phase I testing. The following three specific evaluations of Phase I analytical data were performed: (1) a statistical evaluation of feed sample analytical results, (2) evaluation of percent contaminant removal (Pu-239,240), and (3) evaluation of solids and contaminant mass balances. These evaluations are presented below.

4.2.1 Statistical Evaluation of Feed Sample Analytical Results

For each of the two soil samples and one vegetation sample, the arithmetic mean, standard deviation, and 90 percent confidence intervals were calculated for the feed sample concentrations to assess the variability and homogeneity of the feed sample streams. These statistics were calculated for Pu-239,240. These statistics were calculated using the following formulas:

$$\text{Mean} = \bar{x} = (\sum x_i) / n$$

$$\text{Standard deviation} = s = \sqrt{\frac{n\sum x_i^2 - (\sum x_i)^2}{n\,(n-1)}}$$

$$\text{Upper confidence interval} = (\bar{x}) + \frac{(t_{\alpha/2})(s)}{\sqrt{n}}$$

$$\text{Lower confidence interval} = (\bar{x}) - \frac{(t_{\alpha/2})(s)}{\sqrt{n}}$$

where:

x_i = The concentration of Pu-239,240 measured in an individual replicate chemical characterization analysis of a feed sample during Phase I and Phase II. Four individual replicate analyses were performed for soil sample #1, and 11 such analyses were performed for soil sample #2 and the vegetation sample.

n = The total number of Phase I and Phase II observations (i.e., the number of individual replicate results for each soil or vegetation sample). For soil sample #1, $n = 4$, whereas $n = 11$ for soil sample #2 and the vegetation sample.

$t_{\alpha/2}$ = The Student's t-Distribution for the 90 percent confidence interval for n-1 degrees of freedom ($t_{\alpha/2} = 2.353$ for 3 degrees of freedom [soil sample #1], and $t_{\alpha/2} = 1.812$ for 10 degrees of freedom [soil sample #2 and the vegetation sample]).

Statistics for the feed soil and vegetation samples, encompassing both Phase I and Phase II analytical results, are presented in Table 4.1. Based on the range in values for the standard deviation, the statistics indicate that the highest degree of variability in feed soil results was observed in soil sample #2 and the vegetation sample results. Additional replicate feed samples of soil #2 and the vegetation sample were analyzed to better define this variability. Using a total of 11 replicate feed results, the 90 percent confidence interval of the Pu-239,240 concentration in soil sample #2 ranged from 1000 pCi/g to 1300 pCi/g and from 560 pCi/g to 730 pCi/g in the vegetation sample. For these examples, a standard deviation of 280 pCi/g and 150 pCi/g was calculated for soil sample #2 and the vegetation sample, respectively, relative to mean concentrations of 1200 pCi/g and 640 pCi/g, respectively. Calculation of percent relative standard deviations (%RSDs) based on these values ($s/\bar{x}$ times 100) produce %RSDs of approximately 24 percent for both samples, which is considered acceptable for radiochemical analyses.

The mean feed concentrations calculated from the feed chemical characterization data were compared with treated soil and vegetation data from associated test samples to evaluate Pu-239,240 concentration reduction and calculate Pu-239,240 percent removals and mass balances achieved during the tests. Because Pu-239,240 concentration varied within each feed soil and feed vegetation sample due to sample inhomogeneity, there is potential for component mass balance impacts (i.e., recoveries either greater or less than 100 percent).

4.2.2 Evaluation of Phase I Removal Efficiencies for Plutonium

The percentages of Pu-239,240 removed were calculated for Pu-239,240 for each of the solvent extraction tests conducted in Phase I. The individual percentages of Pu-239,240 removed were calculated using analytical results from feed and treated solid samples as follows:

$$\frac{\text{(Mean Feed Sample Pu-239,240 Concentration)-(Treated Solid Pu-239,240 Concentration)}}{\text{(Mean Feed Sample Pu-239,240 Concentration)}} \times 100 = \text{Pu-239,240 Percent Removal}$$

The calculated results of the Pu-239,240 percent removal are presented below.

4.2.2.1 Soil Sample #1 Pu-239,240 Percent Removal Results for Phase I Testing

The percentages of Pu-239,240 removed during Phase I testing of soil sample #1 are summarized in Table 4.5. The results, based on the mean feed concentration, ranged from 55 to 88 percent removal of Pu-239,240 from soil sample #1. Test 10 demonstrated the maximum mean contaminant removal of 88 percent. However, results of Tests 4, 10, and 15 were all above 80 percent and were equivalent within analytical variance. The parameters used during each of these tests are summarized in Table 2.4.

4.2.2.2 Soil Sample #2 Pu-239,240 Percent Removal Results for Phase I Testing

The percentages of Pu-239,240 removed during Phase I testing of soil sample #2 are presented in Table 4.5. The results, based on the mean feed concentration, ranged from 59 to 86 percent removal

of Pu-239,240 from soil sample #2. Test A demonstrated the maximum mean contaminant removal of 86 percent. However, results of Tests A, B, and C were all above 80 percent and were equivalent within analytical variance. The parameters used during each of these tests are summarized in Table 2.5.

4.2.2.3 Vegetation Sample Pu-239,240 Percent Removal Results for Phase I Testing

The percentages of Pu-239,240 removed during Phase I testing of the vegetation sample are presented in Table 4.5. The results, based on the mean feed concentration, ranged from 51 to 86 percent removal of Pu-239,240 from the sample. Test V-2 demonstrated the maximum mean contaminant removal of 86 percent. The parameters used during each of these tests are summarized in Table 2.6.

4.2.3 Phase I Solids and Pu-239,240 Mass Balances

Mass balance calculations were performed on the basis of Phase I test and analytical results. Mass balance calculations consisted of solids mass balances and Pu-239,240 mass balances for each of the solvent extraction bench-scale tests conducted during Phase I. Solids mass balances for each soil and vegetation sample were computed using the total weight of each feed sample, treated solids samples, in process samples, and nonvolatile reagents used during each solvent extraction test. Data used for calculation of the solids mass balances are presented in Appendix B, Mass Balance Data. The percent mass recovered for each test was calculated as follows:

$$\text{Percent Solids Recovered} = \frac{\text{Mass of effluent solids}}{\text{Mass influent solids}} \times 100\%$$

where:

Mass of effluent solids = Total mass of treated solids recovered after completion of each solvent extraction test

Mass of influent solids = Total mass of feed sample, on a dry basis, and mass of nonvolatile reagents

Contaminant mass balances for Pu-239,240 were conducted using the process and analytical data presented in Appendix A, Analytical Results. The percent contaminant recovery was calculated as follows:

$$\text{Percent Pu-239,240 Recovered} = \frac{\Sigma\,[\text{Conc}_{(fr)} \times \text{Mass}_{(fr)}]}{(\text{Pu-239,240 Mass})_{(in)}} \times 100\%$$

where:

$\text{Pu-239,240 Mass}_{(in)}$ = $\text{Conc}_{(feed)}$ x Mass of feed sample in grams (as received basis)

$\text{Conc}_{(feed)}$ = Concentration of Pu-239,240 in the feed sample, pCi/g (as received basis)

$\text{Conc}_{(fr)}$ = Concentration (as received basis) of Pu-239,240 in the specific process fraction, pCi/g (this includes treated solids, interstage solids, and extract samples).

$\text{Mass}_{(fr)}$ = Total (as received) mass of the specific process fraction, in grams

4.2.3.1 Soil Sample #1 Solids and Contaminant Mass Balance Results for Phase I Testing

Phase I solids and Pu-239,240 mass balance results for soil sample #1 are summarized in Table 4.6. Solids mass balance results for soil sample #1 ranged from 95 to 108 percent recovery. Contaminant mass balance results for soil sample #1 show that the Pu-239,240 recoveries ranged from 41 to 107 percent. With the exception of Test 1, the Pu-239,240 recoveries during Phase I testing of soil sample #1 were within the control limits of 75 to 125 percent used by the laboratory for laboratory control sample (LCS) recoveries. Recoveries outside this range may have been caused by matrix effects, sample inhomogeneity, and/or variations in sample preparation protocols.

4.2.3.2 Soil Sample #2 Solids and Contaminant Mass Balance Results for Phase I Testing

Phase I solids and Pu-239,240 mass balance results for soil sample #2 are summarized in Table 4.6. Solids mass balance results for soil sample #2 ranged from 87 to 96 percent recovery. Contaminant mass balance results for soil sample #2 show that the Pu-239,240 recoveries ranged from 70 to 111 percent. With the exception of Test B, the Pu-239,240 recoveries during Phase I testing of soil

sample #2 were within the control limits of 75 to 125 percent used by the laboratory for LCS recoveries.

4.2.3.3 Vegetation Sample Solids and Contaminant Mass Balance Results for Phase I Testing

Phase I solids and Pu-239,240 mass balance results for the vegetation sample are summarized in Table 4.6. Solids mass balance results for the vegetation sample ranged from 63 to 105 percent recovery. Contaminant mass balance results for the vegetation sample show that the Pu-239,240 recoveries ranged from 110 to 130 percent. These recoveries are near or within the control limit range of 75 to 125 percent recovery used by the laboratory for LCS recoveries. In addition, recoveries of greater than 100 percent for the vegetation sample may be attributed to variability in the total solids analysis of the feed sample.

Table 4.1: Summary of Pu-239,240 Feed Characterization Analytical Results and Statistical Evaluation

	Concentration					
	Chemical Characterization Results Soil Sample #1 (pCi/g)		Chemical Characterization Results Soil Sample #2 (pCi/g)		Chemical Characterization Results Vegetation Sample (pCi/g)	
Concentrations	Phase I	800	Phase II	1500	Phase I	930
		660		1500		680
		690		1200		620
		810		1200		600
			Phase II	710		590
				900		460
				990		620
				940		730
				1200		690
				1100	Phase II	370
				1500		790
Mean		740		1200		640
Standard Deviation		76		280		150
90% Confidence Interval						
Upper Interval		830		1300		730
Lower Interval		650		1000		560

pCi/g Picocuries per gram
% Percent

Table 4.2: Summary of Soil Sample #1 Solids Analytical Results for Phase I Testing

	Results	
	Pu-239,240 (pCi/g)	Total U (μg/g)
Test 1		
Feed*	740	12
5th ext. (final treated) Solids	340	5.4
Test 4		
Feed*	740	12
1st ext. Solids	320	2.7
2nd ext. Solids	190	1.7
3rd est. (final treated) Solids	130	1.0
Test 10		
Feed*	740	12
1st ext. Solids	200	NA
2nd ext. Solids	95	NA
4th ext. (final treated) Solids	86	1.4
Test 15		
Feed*	740	12
1st ext. Solids	120	NA
2nd ext. Solids	6.2	NA
3rd ext. Solids	50	NA
4th ext. (final treated) Solids	95	0.86

ext. Extraction
NA Sample not analyzed for total uranium
pCi/g Picocuries per gram
μg/g Micrograms per gram

* Mean feed concentration (discussed in Section 4.2.1)

Table 4.3: Summary of Soil Sample #2 Solids Analytical Results for Phase I Testing

	Results	
	Pu-239,240 (pCi/g)	**Total U (μg/g)**
Test A		
Feed*	1200	6.2
1st ext. Solids	350	NA
2nd ext. Solids	220	NA
3rd ext. Solids	180	NA
4th ext. (final treated) Solids	170	1.4
Test B		
Feed*	1200	6.2
1st ext. Solids	490	NA
2nd ext. Solids	400	NA
3rd ext. Solids	230	NA
4th ext. (final treated) Solids	230	0.90
Test C		
Feed*	1200	6.2
3rd ext. Solids	170	NA
4th ext. (final treated) Solids	180	NA
Test D		
Feed*	1200	6.2
4th ext. (final treated) Solids	500	1.3

ext. Extraction
NA Sample not analyzed for total uranium
pCi/g Picocuries per gram
μg/g Micrograms per gram

* Mean feed concentration (discussed in Section 4.2.1)

Table 4.4: Summary of Vegetation Solids Analytical Results for Phase I Testing

	Results	
	Pu-239,240 (pCi/g)	**Total U (μg/g)**
Test V-1		
Feed*	640	5.7
3rd ext. Solids	110	NA
4th ext. Solids	81	NA
6th ext. (final treated) Solids	220	0.70
Test V-2		
Feed*	640	5.7
3rd ext. Solids	13	NA
4th ext. Solids	9.0	NA
6th ext. (final treated) Solids	87	0.70
Test V-7		
Feed*	640	5.7
1st ext. Solids	370	NA
2nd ext. (final treated) Solids	310	1.4

ext. Extraction
NA Sample not analyzed for total uranium
pCi/g Picocuries per gram
μg/g Micrograms per gram

* Mean feed concentration (discussed in Section 4.2.1)

Table 4.5: Pu-239,240 Removal During Phase I Testing

Test Number	Number of Extractions	Feed* Concentration (pCi/g)	Treated Solids Concentration (pCi/g)	Percent Removal
Soil Sample #1				
1	5	740	340	55
4	3	740	130	82
10	4	740	86	88
15	4	740	95	87
Soil Sample #2				
A	4	1,200	174	86
B	4	1,200	230	81
C	4	1,200	180	85
D	4	1,200	500	59
Vegetation Sample				
V-1	4	640	220	66
V-2	4	640	87	86
V-7	2	640	310	51

pCi/g Picocuries per gram

* Mean feed concentration (discussed in Section 4.2.1)

Table 4.6: Solids and Pu-239,240 Mass Balance Results for Phase I Testing

Test Number	Solids Recovery (percent)	Pu-239,240 Recovery* (percent)
Soil Sample #1		
1	102	41
4	108	77
10	95	97
15	96	107
Soil Sample #2		
A	96	111
B	87	70
C	94	89
D	93	94
Vegetation Sample		
V-1	63	123
V-2	105	130
V-7	82	110

* Based on mean feed concentrations

5.0 PHASE II TESTING RESULTS AND DISCUSSION

This section presents the results of the Phase II bench-scale testing, as described in Section 2.2, including results for bench-scale solvent extraction tests, sample preparation, evaluation of analytical results, percent contaminant removal calculations, and mass balance calculations. This section also presents results from soil particle size distribution testing and Toxicity Characteristic Leaching Procedure (TCLP) analyses on Phase II test samples.

5.1 Phase II Bench-scale Testing

Phase II solvent extraction tests were performed using the most effective combination of oxidizing/ reducing agents, complexing agents, triethylamine, extraction time, and extraction temperature identified during Phase I testing. Additional extraction stages were conducted during Phase II testing to evaluate the effect of additional extraction stages on removal of plutonium from RFETS soil compared to Phase I.

Phase II analytical results were used to calculate the percent of plutonium removed for each set of process conditions for each soil and vegetation sample tested and provided data for mass balance calculations. Results from the Phase II bench-scale testing (sample preparation and solvent extraction tests) are summarized in the following sections for soil sample #1, soil sample #2, and the vegetation sample.

5.1.1 Results of Feed Sample Preparation

Prior to conducting Phase I testing, soil samples were screened, blended, and split into six separate test samples and two analytical samples, as described in Section 2.1.1. The vegetation sample was prepared, ground, blended, and split into six separate test samples and two analytical samples, as described in Section 2.1.1.3. One of the six Phase I test samples was selected from soil sample #1 and soil sample #2 for Phase II testing. The Phase II test samples were split, blended, and

recombined eight more times to ensure sample homogeneity. Additional samples for feed chemical characterization analyses were withdrawn from the homogenized Phase II test sample and from the remaining Phase I feed samples to better establish feed concentrations and their associated variability, and to provide a mechanism by which to evaluate the effectiveness of the blending process. Review of the Phase I and Phase II chemical characterization soil and vegetation data (Table 4.1) indicates that there were differences in the plutonium concentrations of the feed samples within each soil type and vegetation. The observed variations in the concentrations may be attributable to inherent heterogeneity of the soil and analytical variances (see Section 4.2.1). The results of the feed analyses are presented in Table 4.1 and Appendix A, Analytical Results.

5.1.2 Soil Sample #1 Test Results

The process conditions used during Phase II bench-scale testing of soil sample #1 are summarized as follows:

- The extraction temperature was held constant at 190°F.
- The extraction time was 60 minutes per extraction stage.
- Centrifugation was required.
- The oxidation/reduction and complexing agents tested are presented in Table 2.7.
- The ratio of feed to reagent (i.e., oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, was 8 to 1.
- The ratio of solvent to reagent, expressed on a weight-to-weight basis, varied between 2:1 and 10:1.

The results of Pu-239,240, Am-241, and total uranium analyses conducted on soil sample #1 feed, interstage (i.e., sixth and ninth extraction), and final treated solids from Phase II solvent extraction tests are presented in Table 5.1. The analytical results showed that in Tests 1 and 2, Pu-239,240 was reduced from a mean feed concentration of 740 pCi/g to 88 pCi/g and 83 pCi/g, respectively, in the final treated solids. The concentration of Pu-239,240 in the solids was not significantly reduced after

the initial four extractions. Therefore, conducting four extractions instead of twelve would have yielded similar results. Additional analytical results (i.e., extract solution, extract concentrate) from Phase II, soil sample #1 solvent extraction tests are presented in Appendix A, Analytical Results.

5.1.3 Soil Sample #2 Test Results

The process data collected during Phase II bench-scale testing of soil sample #2 are summarized as follows:

- The extraction temperature was held constant at 190°F.
- The extraction time was 60 minutes per extraction stage.
- Centrifugation was required.
- The oxidation/reduction and complexing agents tested are presented in Table 2.8.
- The ratio of feed to reagent (i.e., oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, was 8 to 1.
- The ratio of solvent to reagent, expressed on a weight-to-weight basis, varied between 2:1 and 10:1.

The results of Pu-239,240, Am-241, and total uranium analyses conducted on soil sample #2 feed, interstage (i.e., sixth extraction, ninth extraction), and final treated solids from Phase II solvent extraction tests are presented in Table 5.2. The analytical results showed that in Tests 1 and 2, Pu-239,240 was reduced from a mean feed concentration of 1200 pCi/g to 100 pCi/g and 355 pCi/g in the final treated solids, respectively. It is not known why the treated solids concentration in Test 2 was significantly higher than the interstage solids concentration for Test 2, although analytical variability is expected to be high in interstage solid results relative to other media (see Section 6.0).

The concentration of plutonium in the solids was not significantly reduced after the initial four extractions. Therefore, conducting four extractions instead of twelve would have yielded similar

results. Additional analytical results (i.e., extraction solution, extraction concentrate) from Phase II, soil sample #2 solvent extraction tests are presented in Appendix A, Analytical Results.

5.1.4 Vegetation Sample Test Results

The process data collected during Phase II bench-scale testing of the RFETS vegetation sample are summarized as follows:

- The extraction temperature was held constant at 190°F.
- The extraction time was 60 minutes per extraction stage.
- Centrifugation was required.
- The oxidation/reduction and complexing agents tested are presented in Table 2.9.
- The ratio of feed to reagent (i.e., oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, was 8 to 1.
- The ratio of solvent to reagent, expressed on a weight-to-weight basis, was 2:1.

The results of Pu-239,240, Am-241, and total uranium analyses conducted on feed, interstage (i.e., sixth extraction, ninth extraction), and final treated solids from Phase II solvent extraction vegetation testing are presented in Table 5.3. The analytical results show that in Test 1, Pu-239,240 was reduced from a mean feed concentration of 640 pCi/g to 23 pCi/g in the final treated solids. The concentration of Pu-239,240 in the solids was not significantly reduced after the initial four extractions. Therefore, conducting four extractions instead of twelve would have yielded similar results. Additional analytical results (i.e., extraction solution, extraction concentrate) from Phase II, vegetation sample solvent extraction tests are presented in Appendix A.

5.2 Evaluation of Phase II Analytical Results

Data evaluation of Phase II analytical results was performed to assist in assessing the overall bench-scale test performance during Phase II testing. The following three specific evaluations of Phase II analytical data were performed: (1) a statistical evaluation of feed analytical results, (2) evaluation of

percent contaminant removal (Pu-239,240), and (3) evaluation of solids and contaminant mass balances. These evaluations are presented in the following sections.

5.2.1 Feed Analysis Evaluation

For each of the two soil samples and one vegetation sample, the arithmetic mean, standard deviation, and 90 percent confidence intervals were calculated for the feed concentrations to assess the variability and homogeneity of the feed sample streams. This statistical analysis was discussed, in detail, in Section 4.2.1.

5.2.2 Evaluation of Removal Efficiencies for Pu-239,240

The percentages of Pu-239,240 removed were calculated for each of the solvent extraction tests conducted in Phase II. The individual percentages of Pu-239,240 removed were calculated using analytical results from feed and treated solid samples as follows:

$$\frac{\text{(Mean Feed Sample Pu-239,240 Concentration)-(Treated Solid Pu-239,240 Concentration)}}{\text{(Mean Feed Sample Pu-239,240 Concentration)}} \times 100 = \text{Pu-239,240 Percent Removal}$$

The calculated results of the Pu-239,240 percent removal are presented below and are based on the mean feed concentrations.

5.2.2.1 Soil Sample #1 Pu-239,240 Percent Removal Results for Phase II Testing

The percentages of Pu-239,240 removed for Phase II testing of soil sample #1 are summarized in Table 5.4. The results for soil sample #1, based on the Phase I mean feed concentration, showed 88 and 89 percent removal of Pu-239,240 for Tests 1 and 2, respectively. The parameters used during each of these tests are summarized in Table 2.7.

5.2.2.2 Soil Sample #2 Pu-239,240 Percent Removal Results for Phase II Testing

The percentages of Pu-239,240 removed for Phase II testing of soil sample #2 are summarized in Table 5.4. The results for soil sample #2, based on the Phase I mean feed concentration, showed 92 and 70 percent removal of Pu-239,240 for Tests 1 and 2, respectively. The parameters used during each of these tests are summarized in Table 2.8. It is not known why the percent removal of Test 2 was significantly below that achieved in a similar test during Phase I testing.

5.2.2.3 Vegetation Sample Pu-239,240 Percent Removal Results for Phase II Testing

The percentage of Pu-239,240 removed for Phase II testing of the vegetation sample is presented in Table 5.4. The results for the vegetation sample, based on the Phase I mean feed concentration, showed a 97 percent removal of Pu-239,240 from the sample. The parameters used during this test are summarized in Table 2.9.

5.2.3 Phase II Solids and Pu-239,240 Mass Balances

Mass balance calculations were performed on the basis of Phase II test and analytical results. Mass balance calculations consisted of solids mass balances and Pu-239,240 mass balances for each of the solvent extraction bench-scale tests conducted in Phase II. These calculations were performed as described for the Phase I tests in Section 4.2.3.

5.2.3.1 Soil Sample #1 Solids and Contaminant Mass Balance Results for Phase II Testing

Solids and Pu-239,240 mass balance results for soil sample #1 are summarized in Table 5.5. Solids mass balance results for soil sample #1 were 109 percent and 92 percent recovery for Tests 1 and 2, respectively. Contaminant mass balance results for soil sample #1 showed that the Pu-239,240 recoveries were 111 percent and 100 percent for Tests 1 and 2, respectively. The Pu-239,240

recoveries for Phase II testing of soil #1 were within the control limits of 75 to 125 percent used by the laboratory for LCS recoveries, and were based on the mean feed concentrations.

5.2.3.2 Soil Sample #2 Solids and Contaminant Mass Balance Results for Phase II Testing

Solids and Pu-239,240 mass balance results for soil sample #2 are summarized in Table 5.5. Solids mass balance results for soil sample #2 were 111 percent and 88 percent recovery for Tests 1 and 2, respectively. Contaminant mass balance results for soil sample #2 showed that the Pu-239,240 recoveries were 92 percent and 103 percent for Tests 1 and 2, respectively. The Pu-239,240 recoveries for Phase II testing of soil #2 were within the control limits of 75 to 125 percent used by the laboratory for LCS recoveries, and were based on the mean feed concentrations.

5.2.3.3 Vegetation Sample Solids and Contaminant Mass Balance Results for Phase II Testing

Solids and Pu-239,240 mass balance results for the vegetation sample are summarized in Table 5.5. Solids mass balance results for the vegetation sample showed 134 percent recovery. Contaminant mass balance results for the vegetation sample showed that the Pu-239,240 recovery was 143 percent. The Pu-239,240 recoveries for Phase II testing of the vegetation sample fell outside the control limits of 75 to 125 percent used by the laboratory for LCS recoveries, and were based on the mean feed concentrations.

5.3 Soil Particle Size and Contaminant Distribution Test Results

A soil particle size and contaminant distribution test was conducted on soil #1 and soil #2 as described in Sections 2.1.4 and 4.1.1. The amount of material captured by each screen and the reported activity of each fraction is presented in Table 5.6. The analytical results for the soil particle size and contaminant distribution test are also presented in Appendix A, Analytical Results.

Results presented in Table 5.6 were used to calculate the distribution of soil weight and total activity among the screen fractions. The approach used to calculate the percent weight and percent total activity for each screen fraction using the soil particle size and contaminant distribution data is as follows:

$$\frac{\left[\begin{array}{l}\text{Total weight (dry) of}\\ \text{soil particles in each}\\ \text{screen fraction (g)}\end{array}\right]}{\left[\begin{array}{l}\text{Total weight (dry) of}\\ \text{the whole sample (g)}\end{array}\right]} \times 100 = \text{Percent of solids in each screen fraction}$$

$$\frac{\left[\begin{array}{l}\text{Total weight}\\ \text{(dry) of each}\\ \text{screen fraction (g)}\end{array}\right] \times \left[\begin{array}{l}\text{Total activity}\\ \text{in each screen}\\ \text{fraction (pCi/g)}\end{array}\right]}{\left[\begin{array}{l}\text{Total weight}\\ \text{(dry) of the}\\ \text{whole sample (g)}\end{array}\right] \times \left[\begin{array}{l}\text{Total activity}\\ \text{of the whole}\\ \text{sample (pCi/g)}\end{array}\right]} \times 100 = \text{Percent of total activity in each screen fraction}$$

Review of the soil sample #1 data indicates that the largest percent of solids, 51 percent, passed through the #40 mesh Tyler screen. Approximately 21 percent of the total activity remained with the solids that passed through the smallest screen, and 68 percent of the total activity was transferred into the aqueous phase. However, only about 5 percent of the total activity originally contained in the feed sample remained in the soil captured on the #5, #8, and #40 screens.

For soil sample #2, the largest percentage of the solids, 65 percent, again passed through the 40 mesh Tyler screen. Approximately 37 percent of the total activity remained with the solids that passed through the smallest screen, and 63 percent of the total activity was transferred into the aqueous phase. However, only about 0.6 percent of the total activity originally contained in the feed sample remained in the soil captured on the #5, #8, and #40 screens.

5.4 Toxicity Characteristic Leaching Procedure Test Results

The Phase II feed material for soil sample # 1 and soil sample #2 was extracted using the Toxicity Characteristic Leaching Procedure (TCLP) in accordance with Federal Register, March 29, 1990. Each

TCLP leachate was analyzed for metals content. The results of this analysis are summarized in Table 5.7. The feed material for both soil samples passed the TCLP test for metals.

The treated solids from each soil test during Phase II testing were extracted using the TCLP procedure described above. Each TCLP leachate was analyzed for metals content. The results of these analyses are summarized in Table 5.8. The treated solids for both soil samples passed the TCLP test for metals. The treated solids for both soil samples passed the TCLP test for metals.

Table 5.1: Summary of Soil Sample #1 Solids Analytical Results for Phase II Testing

	Results		
	Pu-230,240 (pCi/g)	**Total U (μg/g)**	**Am-241 (pCi/g)**
Test 1			
Feed*	740	12	150
6th ext. Solids	42	0.8	8.7
9th ext. Solids	44	0.9	9.4
12th ext. (final treated) Solids	88	0.9	19
Test 2			
Feed*	740	12	150
6th ext. Solids	32	1.1	7.0
9th ext. Solids	35	0.9	7.5
12th ext. (final treated) Solids	83	1.0	16.6

ext. Extraction
pCi/g Picocuries per gram
μg/g Micrograms per gram

* Mean feed concentration (see Section 4.2.1)

Table 5.2: Summary of Soil Sample #2 Solids Analytical Results for Phase II Testing

	Results		
	Pu-230,240 (pCi/g)	Total U (μg/g)	Am-241 (pCi/g)
Test 1			
Feed*	1200	6.2	193
6th ext. Solids	97	2.2	21
9th ext. Solids	108	2.1	22
12th ext. (final treated) Solids	102	2.5	22
Test 2			
Feed*	1200	6.2	193
6th ext. Solids	43	1.1	12
9th ext. Solids	278	1.7	71
12th ext. (final treated) Solids	355	2.7	81

ext. Extraction
pCi/g Picocuries per gram
μg/g Micrograms per gram

* Mean feed concentration (see Section 4.2.1)

Table 5.3: Summary of Vegetation Sample Solids Analytical Results for Phase II Testing

	Results		
	Pu-230,240 (pCi/g)	Total U (μg/g)	Am-241 (pCi/g)
Test 1			
Feed*	640	5.7	130
6th ext. Solids	17	0.74	4.1
9th ext. Solids	19	0.8	4.2
12th ext. (final treated) Solids	23	0.82	5.8

ext. Extraction
pCi/g Picocuries per gram
μg/g Micrograms per gram

* Mean feed concentration (see Section 4.2.1)

Table 5.4: Pu-239,240 Removal for Phase II Testing

Test Number	Number of Extractions	Feed Concentration (pCi/g)	Treated Solids Concentration (pCi/g)	Percent Removal
Soil Sample #1				
1	12	740	88	88
2	12	740	83	89
Soil Sample #2				
1	12	1,200	102	92
2	12	1,200	355	70
Vegetation Sample				
1	12	640	23	97

pCi/g Picocuries per gram

Table 5.5: Solids and Pu-239,240 Mass Balance Results for Phase II Testing

Test Number	Solids Recovery (percent)	Pu-239,240 Recovery (percent)
Soil Sample #1		
1	109	111
2	92	100
Soil Sample #2		
1	111	92
2	88	103
Vegetation Sample		
1	134	143

Table 5.6: Summary of Soil Particle Size and Contaminant Distribution Test Results

	Dry Weight (g)	Percent of Total Dry Weight (g)	Pu-239,240 Activity (pCi/g)	Percent of Total Activity
Soil #1				
Feed soil used	138	100	740 *	100
Sieve fractions collected				
#5 mesh	1.0	0.7	17	0.02
#8 mesh	3.3	2.4	6.8	0.02
#40 mesh	65.5	47	72	4.6
< #40 mesh	69.7	51	310	21
Liquid #	5355	---	13	68
Soil #2				
Feed soil used	133	100	1200 *	100
Sieve fractions collected				
#5 mesh	0.30	0.2	15.8	neg.
#8 mesh	6.2	4.7	10.9	0.04
#40 mesh	40.0	30	23.7	0.5
< #40 mesh	86.9	65	679	37
Liquid #	2965	---	34	63

neg. Negligible

* Mean feed concentration
\# As received basis

Table 5.7: Feed Soil TCLP Leachate Analysis, mg/l

	Sample Result		
Analyte	**Soil No. 1**	**Soil No. 2**	**Regulatory Level, mg/l**
Arsenic	ND (0.2)	ND (0.06)	5
Barium	ND (2.0)	1.0 (0.9)	100
Cadmium	ND (0.01)	ND (0.005)	1
Chromium	ND (0.02)	ND (0.01)	5
Lead	ND (0.1)	ND (0.05)	5
Mercury	ND (0.002)	ND (0.002)	0.2
Selenium	ND (0.2)	ND (0.1)	1
Silver	ND (0.02)	ND (0.01)	5

Values in parentheses indicate detection limit

mg/l Milligrams per liter
ND Not detected

Table 5.8: Treated Solids TCLP Leachate Analyses, mg/l

Analyte	Soil Sample #1 Sample Result Test 1*	Test 2#	Regulatory Level (mg/l)
Arsenic	ND (0.06)	ND (0.06)	5
Barium	ND (0.9)	ND (0.9)	100
Cadmium	ND (0.005)	ND (0.005)	1
Chromium	0.01 (0.01)	ND (0.01)	5
Lead	ND (0.05)	ND (0.05)	5
Mercury	ND (0.002)	ND (0.002)	0.2
Selenium	ND (0.1)	ND (0.1)	1
Silver	ND (0.01)	ND (0.01)	5

Analyte	Soil Sample #2 Sample Result Test 1*	Test 2#	Regulatory Level (mg/l)
Arsenic	ND (0.06)	ND (0.06)	5
Barium	ND (0.9)	ND (0.9)	100
Cadmium	ND (0.005)	ND (0.005)	1
Chromium	0.1 (0.01)	0.02 (0.01)	5
Lead	0.07 (0.05)	0.06 (0.05)	5
Mercury	ND (0.002)	ND (0.002)	0.2
Selenium	ND (0.1)	ND (0.1)	1
Silver	ND (0.01)	ND (0.01)	5

Values in parentheses indicate detection limit

mg/l Milligrams per liter

* Test 1 = Hydrogen Peroxide/Citric Acid Test
\# Test 2 = Sodium Citrate/Sodium Dithionite Test

6.0 QUALITY ASSURANCE AND QUALITY CONTROL SUMMARY

The overall quality of the analytical data generated in support of the bench-scale solvent extraction treatability study was assessed through (1) reviews of analytical QC data and raw data packages from the laboratory and (2) an onsite audit of the laboratory while treatability study samples were being analyzed. The data quality assessment found that the laboratory generally met QA/QC requirements for the study as established in the QAA and in the supporting laboratory SOPs. Radiochemical tracer recoveries were within the QAA control limits of 20 to 105 percent for all but a few treatability samples. In addition, laboratory control sample spikes performed by the laboratory during treatability sample analysis generally met the laboratory's percent recovery criteria of 75 to 125 percent, and analytical splits of treatability study samples generally agreed within a relative percent difference of 25 percent. The laboratory recounted analytical splits that did not meet control criteria, and as time permitted, performed confirmatory reanalyses of these splits when poor precision was indicated.

Although reviews of QA/QC sample results indicated that the treatability study data were of acceptable overall quality, the data quality assessment identified the following concerns regarding error and variability in the analytical results:

1. **Total Propagated Uncertainty.** The laboratory reported a total propagated uncertainty (TPU) for each analytical result based on statistical instrument counting errors, sample counting time, and measurements associated with sample weighing and preparation steps. Due to high target analyte levels, sample counting times had to be reduced for feed samples and many interstage samples in order to preserve instrument integrity. Reductions in sample counting time tended to increase the TPUs associated with the analytical results, which were reported in the range of 15 percent for most of the treatability study samples.

2. **Uncertainties in tracer quantitation.** The high levels of target radionuclides in the feed and interstage samples were not compatible with the laboratory's SOP for spiking of radiochemical tracers. Because the laboratory had historically analyzed only low-level samples, the concentration of the laboratory's tracer spike solutions were not appropriate for use with high-level samples. Even when large amounts of these spike solutions were used, total tracer concentrations remained very low relative to the target radionuclide concentrations in many feed and interstage samples. Observation of the plutonium analyses of such samples indicated that the large Pu-239 target analyte peak was capable of easily overwhelming the

adjacent tracer peak as the sample was counted. Although the laboratory took care to resolve and quantitate the tracer peak as accurately as possible (e.g., by decreasing sample counting time), a potential for error was assessed to exist in tracer quantification due to the proximity of the large Pu-239 peak. Since tracer recoveries are used to correct the target analyte results, errors in tracer quantification translate into an increased potential uncertainty in the analytical results for the feed and interstage samples that might not be reflected in the TPU.

3. **Weighing and sample homogeneity.** Due to the high activity of the feed and interstage samples, sample aliquots were reduced to 0.5 gram for plutonium analysis from the 2.0 gram aliquot specified in the laboratory's SOP. Sample heterogeneity may affect analytical precision for these small sample aliquots. In addition, weighing errors as high as 6 percent were observed during the audit due to balance fluctuations and variations in sample mass measurements allowed by the analyst. These errors were higher than the weighing error assumed for calculation of TPU, and could indicate additional unquantified uncertainty in the final analytical results.

   It was observed during the audit that the laboratory's sample grinding procedure was sufficient for soil samples, resulting in finely-ground, homogeneous analytical samples. However, vegetation samples were more difficult to homogenize; significant variation in particle size was observed even after prolonged grinding. Thus, sample heterogeneity might contribute to analytical variability in the vegetation sample results.

4. **Preparation of vegetation samples.** Whereas a total dissolution method was used to prepare soil samples, a wet ashing method was used by the laboratory to prepare vegetation samples. The wet ashing method involved multiple digestions of the samples with nitric acid to leach the target radionuclides and remove organics. It was observed during the audit that significant insoluble solids remained after the wet ashing step that were filtered away from the samples before proceeding with sample cleanup and analysis. Thus, it is possible that vegetation sample results are biased low due to the loss of target radionuclides trapped in this insoluble solid. Loss of target radionuclides is consistent with the low tracer recoveries observed for vegetation samples, which ranged from 20 to 40 percent compared to 50 to 80 percent for soil samples. It is recommended that the laboratory reanalyze a number of vegetation samples by the soil method (i.e., by the total dissolution method) for comparison to results obtained by the wet ash method.

5. **Analyte loss during cleanup.** Cleanup procedures for both soil and vegetation samples analyzed for plutonium included the use of an ion exchange column to remove extraneous actinides and salts. Analyses of column eluants requested during the audit indicated that premature column breakthrough may have occurred during cleanup of the feed samples. This breakthrough apparently resulted in loss of plutonium from the sample while on the ion exchange column, hence analytical results for these samples may be biased low. Column breakthrough during sample cleanup and the approximate magnitude of the resulting bias in feed sample data is currently under further investigation at the laboratory.

The above findings of the data quality assessment for the bench-scale solvent extraction treatability study are presented to document potential sources of variability and bias in the analytical data. As such, these findings are presented to caution and assist data users in their interpretation and use of

the analytical results. The laboratory audit and data package reviews indicated that the potential for imprecision and bias is greatest in the results reported for high-level samples such as feed and interstage treated samples. Specifically, the potential for low bias in the feed sample results due to findings 4 and 5 above indicate that Pu-239,240 percent removals calculated in Sections 4.0 and 5.0 may also be biased low, whereas Pu-239,240 mass recoveries may be biased high. It is important to note, however, no findings of the data quality assessment required the rejection of treatability study results.

7.0 SUMMARY OF RESULTS AND RECOMMENDATIONS

The results of the Phase I bench-scale tests, as summarized in Table 7.1, indicate that a significant removal of Pu-239,240 from RFP soil and vegetation was achieved during Phase I testing. The Pu-239,240 removal for the tests with the most favorable results, following four extractions, was 88 percent for soil sample #1, 86 percent for soil sample #2, and 86 percent for the vegetation sample. Pu-239,240 and solids mass balance results, summarized in Table 7.1, support the Pu-239,240 removal results. Solids recovery ranged from 95 to 105 percent and Pu-239,240 recovery ranged from 97 to 130 percent.

The results of the Phase II bench-scale tests, as summarized in Table 7.2, indicate that removal of Pu-239,240 from RFETS soil and vegetation during Phase II testing was similar to that achieved during Phase I testing. This indicates that the majority of the plutonium removal occurred in the first three to four extraction stages. The Pu-239,240 removal for the Phase II tests with the most favorable results was 89 percent for soil sample #1, 92 percent for soil sample #2, and 96 percent for the vegetation sample, based on the mean feed concentrations. Pu-239,240 and solids mass balance results, summarized in Table 7.2, support the Pu-239,240 removal results. Solids recovery ranged from 88 to 134 percent and Pu-239,240 recovery ranged from 81 to 148 percent.

Based on the results of the Phase II bench-scale solvent extraction testing, it is evident that virtually all plutonium extraction took place during the first four extraction stages and the final eight extraction stages accomplished only limited additional extraction. Removal of residual plutonium from the treated solids to below the TSBs would require further optimization testing to lower the liquid-to-feed ratio and to identify the optimum extraction parameters.

Results of the soil particle size and contaminant distribution testing, presented in Section 5.3, demonstrated that virtually all of the plutonium contamination contained in the soil samples exists in the fraction that was small enough to pass through a 40 mesh screen (only 5 percent of the contamination in soil sample #1 and 0.6 percent of the contamination in soil sample #2 exists in particle sizes larger than 40 mesh). In addition, more than 60 percent of the soil contamination was transported into the aqueous phase during the test.

Table 7.1: Summary of Phase I Bench-scale Test Results

Sample	Reagents Used (Redox/Complexing/Other)	Number of Extractions	Pu-239,240 Removal (percent)	Pu-239,240 Recovery (percent)	Solids Recovery (percent)
Soil sample #1	$Na_2S_2O_4/Na_3C_6H_5O_7$/---	4	88	97	95
Soil sample #2	$Na_2S_2O_4/Na_3C_6H_5O_7$/---	4	86	111	96
Vegetation sample	$Na_2S_2O_4/Na_3C_6H_5O_7$/TEA	4	86	130	105

TEA Triethylamine
Redox Reducing/oxidizing agent
--- No other reagent used

Table 7.2: Summary of Phase II Bench-scale Test Results

Sample	Reagents Used (Redox/Complexing/Other)	Number of Extractions	Pu-239,240 Removal (percent)	Pu-239,240 Removal Required to Meet TSBs (percent)	Pu-239,240 Recovery (percent)	Solids Recovery (percent)
Soil #1						
Test #1	$H_2O_2/C_6H_8O_7$/TEA	12	88	99.5	111	109
Test #2	$Na_2S_2O_4/Na_3C_6H_5O_7$/TEA	12	89	99.5	100	92
Soil #2						
Test #1	$H_2O_2/C_6H_8O_7$/TEA	12	92	99.7	92	111
Test #2	$Na_2S_2O_4/Na_3C_6H_5O_7$/TEA	12	70	99.7	103	88
Vegetation	$Na_2S_2O_4/Na_3C_6H_5O_7$/TEA	12	97	99.4	143	134

TEA Triethylamine
Redox Reducing/oxidizing agent

8.0 ACRONYMS

Am	Americium
ARAR	Applicable or Relevant and Appropriate Requirement
ATI	Analytical Technologies, Inc.
CFR	Code of Federal Regulations
cm	Centimeter
COC	Contaminant of concern
CTR	Contractors Technical Representative
FS	Feasibility Study
HASP	Health and Safety Plan
kg	Kilogram
LCS	Laboratory control sample
M	Molar
OU	Operable Unit
pCi/g	Picocuries per gram
PPE	Personal protective equipment
Pu-239,240	Total concentration of plutonium 239 and plutonium 240
PRG	Preliminary Remediation Goals
QA	Quality assurance
QAA	Quality Assurance Addendum
QAPP	Quality Assurance Project Plan
QC	Quality control
RCC	Resources Conservation Company
RCRA	Resource Conservation and Recovery Act
RFETS	Rocky Flats Environmental Technology Site

SHMP	Sodium hexametaphosphate
SOP	Standard operating procedure
TCLP	Toxicity Characteristic Leaching Procedure
TSB	Treatability Study Benchmark
TPU	Total propagated uncertainty
U	Uranium
U.S.	United States
°F	Degrees Fahrenheit
μg/g	Micrograms per gram

Appendix A

ANALYTICAL RESULTS

PHASE I

Analyses Results

Sample ID	Description	Pu-239/240, pCi/g	Pu-238, pCi/g	KPA U, ug/g	Gross a, pCi/g	Gross b, pCi/g	Am-241, pCi/g	O&G, mg/kg
Soil Sample Number One								
94-07-177								
-02	Left Tree Side Split	556.5	9.595	8.12				28000
-03	Left Tree Side Split	447.93	7.171	8.01	572.38	29.72		
-04	Left Tree Side Split						144	
redo of -03	Redo after Grinding	798.97	12.51					
94-07-178								
-02	Feed Sample for Quick Gross Alpha				660.49	36.48		
-03	Test 1 Treated Solids	335.79	5.667	5.354				
-04	Test 1 Organic Concentrate	6.9	< 0.132	0.068				
-05	Test 4, 2 extract soln	51.985	0.789					
-06	Test 4, 3 extract soln, heavy phase	235.27	3.835	3.614				
-07	Test 4, 3 solids	321.59	5.324	2.659				
-08	Test 4, 3 sump concentrate	1.075	0.016					
-09	Test 4, 5 extract soln	26.584	0.384					
-10	Test 4, 6 solids	188.87	3.198	1.662				
-11	Test 4, 6 extract soln, heavy phase	87.308	1.445					
-12	Test 4, 10 extract soln, heavy phase	34.282	0.599	0.181				
-13	Test 5, 2 extract soln	72.949	1.1					
-14	Test 5, 3 extract soln, heavy phase	146	2.37					
-15	Test 4, Treated solids	134	2.18	1.011				
94-07-179								
-02	Test 6, 2 extract soln	19.144	0.288					
-03	Test 6, treated solids	952.57	15.55	2.663				
-04	Test 10, 1 extract soln, ovnt mix	66.731	1.06					
-05	Test 10, 1 solids	205.4	3.694					
-06	Test 10, 2 extract soln	6.383	0.107					
-07	Test 10, 2 solids	94.788	1.198					
-08	Test 10, 4 extract soln	0.403	0.006					
-09	Test 10, Treated solids	86.4	1.63	1.371				
-10	Test 10, 3 extract soln	0.848	0.017					
-11	Test 10, final heavy phase concentrate	84.4	1.37					
-14	Test 15, 1 solids	116.55	1.977					
-15	Test 15, 2 extract soln	11.944	0.186					
-16	Test 15, 2 solids	6.167	< 0.405					
-17	Test 15, 3 extract soln	2.018	0.022					
-18	Test 15, 3 solids	50.126	0.784					
-19	Test 15, 4 extract soln	1.358	< 0.034					
-20	Test 15, Treated solids	94.7	1.72	0.86				
-21	Test 15, concentrated extract soln	283	4.52					
-23	Test 15, 1 extract soln	68.7	1.06					
94-07-184								
-02	Right side tree split	526.41	8.829	8.34				
-03	Right side tree split	501.65	7.018	8.265				
Redo -02	redo	664.01	10.414					
Redo -03	redo	748.76	12.955					

Sample ID	Description	Pu-239/240, pCi/g	Pu-238, pCi/g	KPA U, ug/g	Gross a, pCi/g	Gross b, pCi/g	Am-241, pCi/g	O&G, mg/kg
Soil Sample Number Two								
94-08-049								
-02	Left side tree split	1530.2	23.865	6.634	1127.5	51.13		1200
-03	left side tree split	1514.3	24.27	6.435				
94-08-050								
-02	Test A, 1 solids	353.09	5.914					
-03	Test A, 1 extract soln	109.73	1.654					
-04	Test A, 2 solids	219.29	4.284					
-05	Test A, 2 extract soln	15.806	0.225					
-06	Test A, 3 solids	182.73	3.414					
-07	Test A, 3 extract soln	4.741	0.078					
-08	Test A, Treated solids	174	3.35	1.415				
-09	Test A, 4 extract soln	1.38	0.023					
-10	Test B, 1 extract soln	35.9	0.6					
-11	Test B, 1 solids	486.59	7.401					
-12	Test B, 2 extract soln	22.465	0.332					
-13	Test B, 2 solids	406.59	6.995					
-14	Test B, 3 extract soln	5.558	0.077					
-15	Test B, 3 solids	227.84	4.306					
-16	Test B, 4 extract soln	4.223	0.073					
-17	Test B, Treated solids	229	4.17	0.895				
-19	Test C, 3 solids	172.14	3.108					
-20	Test C, Treated solids	184	3.39	1.069				
-21	Test C, composite extract soln	29.762	0.467					
-22	Test D, 1st composite extract soln	0.034	< 0.085					
-23	Test D, 2nd composite extract soln	56.8	0.91					
-24	Test D, treated solids	496	7.59	1.269				
94-08-056								
-02	Right side tree split	1200.1	19.919	7.195				
-03	Right side tree split	1152.5	19.072	6.52			251.86	

Sample ID	Description	Pu-239/240, pCi/g	Pu-238, pCi/g	KPA U, ug/g	Gross a, pCi/g	Gross b, pCi/g	Am-241, pCi/g	O&G, mg/kg
Vegetation Sample								
94-08-296								
-01	Homogenized Feed						74.929	
-02	Left side tree split	680.78	10.766	5.935				
-03	Left side tree split	598.8	9.432	5.82				
94-08-297								
-02	Test V1, 1 extract soln	21.2	0.32					
-03	Test V1, 2 extract soln	7.76	0.1					
-04	Test V1, 3 extract soln	3.26	0.064					
-05	Test V1, 4 extract soln	1.06	< 0.033					
-06	Test V1, 6 extract soln	< 0.021	< 0.057					
-07	Test V1, 3 solids	109.36	< 2.049					
-08	Test V1, 4 solids	81.036	1.288					
-09	Test V1, Treated solids	221	3.58	0.695				
-10	Test V2, 1 extract soln	31.5	0.52					
-11	Test V2, 2 extract soln	5.67	0.078					
-12	Test V2, 3 extract soln	1.35	< 0.035					
-13	Test V2, 4 extract soln	0.25	< 0.024					
-14	Test V2, 6 extract soln	< 0.023	< 0.052					
-15	Test V2, 3 solids	13.162	< 0.887					
-16	Test V2, 4 solids	8.991	< 1.091					
-17	Test V2, Treated solids	87.2	1.68	0.875				
94-08-298								
-02	Test V7, 1 solids	366.05	5.865					
-03	Test V7, 1 extract soln	16.3	0.261					
-04	Test V7, 2 extract soln	6	0.093					
-05	Test V7, Treated solids	311.89	5.09	1.434				
94-08-299								
-02	Right side tree split	462.78	7.261	5.59	719.72	27.47		
-03	Right side tree split	724.59	11.099	5.41				

PHASE II

Sample ID	Description	Test No.	Analyses Results Pu-239/240,	Pu-238, pCi/g	Am-241, pCi/g	KPA U ug/g
Soil Sample Number One						
94-10-169						
-01	Untreated Feed	1&2	688	11.5	152.02	8.54
-02	6th Extraction Solids	1	41.8	0.757	8.712	0.75
-03	9th Extraction Solids	1	44.2	0.764	9.4248	0.9
-04	Composite Extract 1-6	1	14.3	0.242	2.421	166.53
-05	Composite Extract 7-12	1	0.146	< 0.014	0.035	1.63
-06	Treated Solids	1	88.3	1.63	18.968	0.89
-07	6th Extraction Solids	2	32.4	0.577	7.054	1.12
-08	9th Extraction Solids	2	35.4	0.651	7.464	0.93
-09	Composite Extract 1-6	2	13.3	0.236	2.296	183.61
-10	Composite Extract 7-12	2	0.335	< 0.022	0.085	1.78
-11	Treated Solids	2	83.3	1.46	16.572	0.99
-12	Recovered Water	1	0.605	< 0.022	0.132	8.42
-13	Concentrated Contaminant	1	153	2.63	27.829	1.43
-14	Recovered Water	2	0.041	< 0.048	< 0.011	8.01
-15	Concentrated Contaminant	2	42.7	0.734	7.582	57.59
Soil Sample Number Two						
94-10-233						
-01	Untreated Feed	1&2	707/895	11.7	192.68	6.36
-02	6th Extraction Solids	1	97.1	1.72	20.658	2.17
-03	9th Extraction Solids	1	108	1.97	21.297	2.059
-04	Treated Solids	1	102	1.84	22.408	2.48
-05	Composite Extract 1-6	1	18.6	0.317	3.254	0.103
-06	Composite Extract 7-12	1	0.298	< 0.005		0.001
-07	Recovered Water	1	0.113	< 0.011	0.023	0.001
-08	Concentrated Contaminant	1	26.4	0.441	5.788	0.083
-09	6th Extraction Solids	2	42.8	< 0.736	12.333	1.11
-10	9th Extraction Solids	2	278	4.68	71.315	1.72
-11	Treated Solids	2	355	6.14	80.73	2.72
-12	Composite Extract 1-6	2	16.6	0.256	2.912	0.103
-13	Composite Extract 7-12	2	0.911	0.015	0.379	0.003
-14	Recovered Water	2	0.69	0.013	0.131	0.016
-15	Concentrated Contaminant	2	37.8	0.562	6.978	0.182
Vegetation Sample						
94-10-298						
-01	Feed	1	370	5.68	130.19	5.48
-02	6th Extraction Solids	1	16.6	< 0.492	4.109	0.74
-03	9th Extraction Solids	1	19.2	0.392	4.238	0.8
-04	Composite Extract 1-6	1	5.21	0.072	1.007	0.019
-05	Composite Extract 7-12	1	0.113	< 0.012	< 0.040	0
-06	Treated Solids	1	22.6	0.381	5.829	0.82
-07	Recovered Water	1	0.234	< 0.08	0.074	0.001
-08	Concentrated Contaminant	1	7.29	0.111	1.57	0.016

		Isotopic Uranium, pCi/g U-234	U-235	U-238	O&G mg/kg
94-10-169-01	Feed	1.09	0.09	3.09	
94-10-233-01	Feed	1.07	< 0.08	2.41	
94-10-298-01	Feed	0.52	< 0.07	1.71	
94-10-233-11	Treated Solids				618

Feed Analyses

All results Pu-239/240, Dry Basis, pCi/g

Description	Sample No.	Result
SOIL NO 1		
PHASE I		
Anal. Split	94-07-177-03	799
Anal. Split	94-07-184-02	664
PHASE II		
Feed	94-10-169-01	688
Feed	94-10-169-01r	812
Mean Conc.		741
SOIL NO 2		
PHASE I		
Anal. Split	94-08-049-02	1530
Anal. Split	94-08-049-03	1514
Anal. Split	94-08-056-02	1200
Anal. Split	94-08-056-03	1152
PHASE II		
Feed	94-10-233-01	707
Feed	94-10-233-01	895
Anal. Split	94-08-049-01	988
Feed Split	94-08-052-01	944
Feed Split	94-08-053-01	1170
Feed Split	94-08-054-01	1090
Feed Split	94-08-051-01	1530
Mean Conc.		1156

Description	Sample No.	Result
VEGETATION		
PHASE I		
Feed Split	94-08-296-01	926
Anal. Split	94-08-296-02	681
Anal. Split	94-08-296-02	616
Anal. Split	94-08-296-03	599
Anal. Split	94-08-296-03	590
Anal. Split	94-08-299-02	463
Anal. Split	94-08-299-02	617
Anal. Split	94-08-299-03	725
Anal. Split	94-08-299-03	691
PHASE II		
Feed	94-10-298-01	370
Feed	94-10-298-01	790
Mean Conc.		643

TOTAL METALS

Sample 94-07-184-03, Soil No. 1

Analyte	Concentration, mg/kg
Aluminum	5,300
Barium	50
Calcium	1,800
Copper	16
Iron	10,000
Lead	20
Magnesium	1,900
Manganese	190
Potassium	2,100
Sodium	60
Silicon	2,100
Zinc	45

Sample 94-08-056-02, Soil No. 2

Analyte	Concentration, mg/kg
Aluminum	7,600
Barium	80
Calcium	4,600
Iron	15,000
Lead	64
Magnesium	2,700
Manganese	260
Potassium	2,300
Sodium	7,900
Silicon	130
Zinc	62

All results reported on an "as received" basis.

SOIL SIEVE TEST							
					Activity per fraction		
Solids Balance				Pu-239,240	Total Activity	Pu Recovery	
Soil #1		%/fraction		pCi/g	pCi	%	
Soil Input, g	138.0			740	102120		
Soil Recovery, g							
> #5 sieve	1.0	0.7%		16.7	16.7	0.0%	
> #8 sieve	3.3	2.4%		6.8	22.44	0.0%	
> #40 sieve	65.5	47.5%		72	4716	4.6%	
< #40 sieve	69.7	50.5%		312	21746.4	21.3%	
Total Recovered	139.5					26%	in solids
% Solids Recovery	101%						
Water Recovered, g	5355.2			13.1	70153.12	69%	in water
					Total Pu Recovery =	95%	
				Pu-239,240	Total Activity	Pu Recovery	
Soil #2		%/fraction		pCi/g	pCi	%	
Soil Input	133.5			1200	160200		
Soil Recovery							
> #5 sieve	0.3	0.2%		15.8	4.74	0.0%	
> #8 sieve	6.2	4.7%		10.9	67.689	0.0%	
> #40 sieve	40.0	29.9%		23.7	946.815	0.6%	
< #40 sieve	86.9	65.1%		679	59005.1	36.8%	
Total Recovered	133.4					37%	in solids
% Solids Recovery	100%						
Water Recovered, g	2964.7			34	100799.8	63%	in water
					Total Pu Recovery =	100%	

Appendix B

MASS BALANCE DATA

PHASE I

Test 1 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	900.72	740	666532.8
Total Input			666532.8
ACTIVITY OUTPUT			
Treated Solids	802.8	335.79	269572
Organic Concentrate	132.4	6.9	914
Samples	116.5	not anal.	NA
Total Output			270486
TOTAL INPUT	666532.8		
TOTAL OUTPUT	270486		
PERCENT RECOVERY	40.6%	Percent Removal	54.6%

Test 4 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	89.9	740	66526
Total Input			66526
ACTIVITY OUTPUT			
2 Extract sol'n	41.8	51.985	2173
3 Heavy Phase	84.4	235.27	19857
3 Concentrate	158.6	1.075	170
3 Solids	5.2	321.59	1672
5 Extract sol'n	39.9	26.584	1061
6 Extract sol'n	107.2	87.308	9359
6 Solids	5.4	188.87	1020
10 Extract sol'n	155	34.282	5314
Treated Solids	78.3	134	10492
Total Output			51118
TOTAL INPUT	66526		
TOTAL OUTPUT	51118		
PERCENT RECOVERY	76.8%	Percent Removal	81.9%

Test 10 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	90	740	66600
Total Input			66600
ACTIVITY OUTPUT			
1 Extract sol'n	758	66.731	50582
1 Solids	8.3	205.4	1705
2 Extract sol'n	710.2	6.383	4533
2 Solids	3.5	94.788	332
3 Extract sol'n	683.8	0.848	580
4 Extract sol'n	693.3	0.403	279
Treated Solids	75.2	86.4	6497
Total Output			64508
TOTAL INPUT	66600		
TOTAL OUTPUT	64508		
PERCENT RECOVERY	96.9%	Percent Removal	88.3%

Test 15 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	90	740	66600
Total Input			66600
ACTIVITY OUTPUT			
1 Extract sol'n	756.5	68.7	51972
1 Solids	3.2	116.55	373
2 Extract sol'n	760.7	11.944	9086
2 Solids	3	6.167	19
3 Extract sol'n	707.1	2.018	1427
3 Solids	4.5	50.126	226
4 Extract sol'n	728	1.358	989
Treated Solids	75.8	94.7	7178
Total Output			71268
TOTAL INPUT	66600		
TOTAL OUTPUT	71268		
PERCENT RECOVERY	107.0%	Percent Removal	87.2%

Test A Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	87.5	1200	105000
Total Input			105000
ACTIVITY OUTPUT			
1 Extract sol'n	758.9	109.73	83274
1 Solids	5.1	353.09	1801
2 Extract sol'n	781.6	15.806	12354
2 Solids	4.6	219.29	1009
3 Extract sol'n	705.3	4.741	3344
3 Solids	4.3	182.73	786
4 Extract sol'n	663.1	1.38	915
Treated Solids	72.1	174	12545
Total Output			116028
TOTAL INPUT	105000		
TOTAL OUTPUT	116028		
PERCENT RECOVERY	110.5%	Percent Removal	85.5%

Test B Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	87.5	1200	105000
Total Input			105000
ACTIVITY OUTPUT			
1 Extract sol'n	734.1	39.745	29177
1 Solids	4.8	486.59	2336
2 Extract sol'n	753.9	22.465	16936
2 Solids	7.1	406.59	2887
3 Extract sol'n	710.3	5.558	3948
3 Solids	6.9	227.84	1572
4 Extract sol'n	635.1	4.223	2682
4 Fines	1.9	879.95	1672
Treated Solids	54.8	229	12549
Total Output			73759
TOTAL INPUT	105000		
TOTAL OUTPUT	73759		
PERCENT RECOVERY	70.2%	Percent Removal	80.9%

Test C Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	87.5	1200	105000
Total Input			105000
ACTIVITY OUTPUT			
Composite Extract sol'n	2714.6	29.762	80792
1 & 2 Solids	14.2		0
3 Solids	6.3	172.14	1084
Treated Solids	64.2	184	11813
Total Output			93689
TOTAL INPUT	105000		
TOTAL OUTPUT	93689		
PERCENT RECOVERY	89.2%	Percent Removal	84.7%

Test D Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	8.75	1200	10500
Total Input			10500
ACTIVITY OUTPUT			
Comp. Extract sol'n 1	96.1	0.034	3
Comp. Extract sol'n 2	100.4	56.8	5703
Treated Solids	8.4	496	4166
Total Output			9872
TOTAL INPUT	10500		
TOTAL OUTPUT	9872		
PERCENT RECOVERY	94.0%	Percent Removal	58.7%

Test V1 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	16.7	640	10688
Total Input			10688
ACTIVITY OUTPUT			
1 Extract sol'n	294.5	21.2	6243
2 Extract sol'n	396.5	7.76	3077
3 Extract sol'n	402.6	3.26	1312
3 Solids	0.5	109.36	55
4 Extract sol'n	385.2	1.06	408
4 Solids	1.3	81.036	105
5 Extract sol'n	217.6	NA	
6 Extract sol'n	204.2	0	0
Treated Solids	8.8	221	1945
Total Output			13146
TOTAL INPUT	10688		
TOTAL OUTPUT	13146		
PERCENT RECOVERY	123.0%	Percent Removal	65.5%

Test V2 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	16.7	640	10688
Total Input			10688
ACTIVITY OUTPUT			
1 Extract sol'n	307.5	31.5	9686
2 Extract sol'n	387.1	5.67	2195
3 Extract sol'n	369.2	1.35	498
3 Solids	1.2	13.162	16
4 Extract sol'n	372.5	0.25	93
4 Solids	2	8.991	18
5 Extract sol'n	215.6	NA	
6 Extract sol'n	198.3	0	0
Treated Solids	15.5	87.2	1352
Total Output			13858
TOTAL INPUT	10688		
TOTAL OUTPUT	13858		
PERCENT RECOVERY	129.7%	Percent Removal	86.4%

Test V7 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	16.7	640	10688
Total Input			10688
ACTIVITY OUTPUT			
1 Extract sol'n	325.2	16.3	5301
1 Solids	1.6	366.05	586
2 Extract sol'n	356.9	6	2141
2 Solids	12.1	311.89	3774
Total Output			11802
TOTAL INPUT	10688		
TOTAL OUTPUT	11802		
PERCENT RECOVERY	110.4%	Percent Removal	51.3%

PHASE II

Soil No. 1, Citrate/Dithionite Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	184	740	136160
Total Input			136160
ACTIVITY OUTPUT			
Treated Solids	159.2	88.3	14057
6th Extraction Solids	7	41.8	293
9th Extraction Solids	8.7	44.2	385
Composite Extract (1-6)	9446.7	14.3	135088
Composite Extract (7-12)	9640.1	0.146	1407
Total Output			151230
TOTAL INPUT	136160		
TOTAL OUTPUT	151230		
PERCENT RECOVERY	111.1%	Percent Removal	88.1%

Soil No. 1 Total Solids = 92%

Soil No. 1, Citric Acid/Peroxide Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	184	740	136160
Total Input			136160
ACTIVITY OUTPUT			
Treated Solids	144.4	83.3	12029
6th Extraction Solids	9	32.4	292
9th Extraction Solids	10.8	35.4	382
Composite Extract (1-6)	9081.3	13.3	120781
Composite Extract (7-12)	9242.8	0.335	3096
Total Output			136580
TOTAL INPUT	136160		
TOTAL OUTPUT	136580		
PERCENT RECOVERY	100.3%	Percent Removal	88.7%

Soil No. 1 Total Solids = 92%

Soil No. 2, Citrate/Dithionite Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi	
ACTIVITY INPUT				
Feed	178	1200	213600	Soil No. 2 Total Solids = 89%
Total Input			213600	
ACTIVITY OUTPUT				
Treated Solids	148.1	102	15106	
6th Extraction Solids	6.6	97.1	641	
9th Extraction Solids	10.3	108	1112	
Composite Extract (1-6)	9459.7	18.6	175950	
Composite Extract (7-12)	9585.8	0.298	2857	
Total Output			195666	
TOTAL INPUT	213600			
TOTAL OUTPUT	195666			
PERCENT RECOVERY	91.6%	Percent Removal	91.5%	

Soil No. 2, Citric Acid/Peroxide Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi	
ACTIVITY INPUT				
Feed	178	1200	213600	Soil No. 2 Total Solids = 89%
Total Input			213600	
ACTIVITY OUTPUT				
Treated Solids	133.3	355	47322	
6th Extraction Solids	5.5	42.8	235	
9th Extraction Solids	7.3	278	2029	
Composite Extract (1-6)	9671.5	16.6	160547	
Composite Extract (7-12)	9652.4	0.911	8793	
Total Output			218927	
TOTAL INPUT	213600			
TOTAL OUTPUT	218927			
PERCENT RECOVERY	102.5%	Percent Removal	70.4%	

Vegetation, Citrate/Dithionite Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	29	640	18560
Total Input			18560
ACTIVITY OUTPUT			
Treated Solids	25	22.6	565
6th Extraction Solids	1.5	16.6	25
9th Extraction Solids	2.2	19.2	42
Composite Extract (1-6)	4876.8	5.21	25408
Composite Extract (7-12)	4891.2	0.113	553
Total Output			26593
TOTAL INPUT	18560		
TOTAL OUTPUT	26593		
PERCENT RECOVERY	143.3%	Percent Removal	96.5%

Vegetation Total Solids = 29%

Test 1 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	1000.8	90.0%	900.72
Total Input			900.72
SOLIDS OUTPUT			
2 Solids	76.4	100.0%	76
4 Solids	40.1	100.0%	40
Treated Solids	802.8	100.0%	803
Total Output			919
TOTAL INPUT	900.72		
TOTAL OUTPUT	919		
PERCENT RECOVERY	102.1%		

Test 4 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	90.0%	90
Total Input			90
SOLIDS OUTPUT			
1 Solids	5.2	100.0%	5
2 Solids	5.4	100.0%	5
Reagent Solids	7.86	100.0%	8
Treated Solids	78.3	100.0%	78
Total Output			97
TOTAL INPUT	90		
TOTAL OUTPUT	97		
PERCENT RECOVERY	107.5%		

Test 10 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	90.0%	90.0
Reagent Heel	35	4.3%	1.5
Total Input			91.5
SOLIDS OUTPUT			
1 Solids	8.3	100.0%	8
2 Solids	3.5	100.0%	4
Treated Solids	75.2	100.0%	75
Total Output			87

TOTAL INPUT	92
TOTAL OUTPUT	87
PERCENT RECOVERY	95.1%

Reagent Heel

0.1M Sodium Dithionite >>>>>>> FW = 174.11g/M
TS = 17.4g/l (0.0174g/ml)

0.1M Sodium Citrate
FW = 258.07g/M
TS = 25.81g/l (0.0258g/ml)

Test 15 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	90.0%	90.0
Reagent Heel	23.7	1.2%	0.3
Total Input			90.3
SOLIDS OUTPUT			
1 Solids	3.2	100.0%	3
2 Solids	3	100.0%	3
3 Solids	4.5	100.0%	5
Treated Solids	75.8	100.0%	76
Total Output			87

TOTAL INPUT	90
TOTAL OUTPUT	87
PERCENT RECOVERY	95.8%

Reagent Heel

0.1M Citric Acid >>>>>>> Used 11.5g to 1 l DIW
TS = 11.5g/l (0.0115g/ml)

Test A Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	87.5%	87.5
Reagent Heel	54.1	4.3%	2.3
Total Input			89.8
SOLIDS OUTPUT			
1 Solids	5.1	100.0%	5
2 Solids	4.6	100.0%	5
3 Solids	4.3	100.0%	4
Treated Solids	69.1	100.0%	69
Residual	3	100.0%	3
Total Output			86
TOTAL INPUT	90		
TOTAL OUTPUT	86		
PERCENT RECOVERY	95.8%		

Test B Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	87.5%	87.5
Reagent Heel	29	1.2%	0.3
Total Input			87.8
SOLIDS OUTPUT			
1 Solids	4.8	100.0%	5
2 Solids	7.1	100.0%	7
3 Solids	6.9	100.0%	7
Treated Solids	56.7	100.0%	57
Centrate Solids	635.1	0.2%	1
Total Output			77
TOTAL INPUT	88		
TOTAL OUTPUT	77		
PERCENT RECOVERY	87.4%		

Test C Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	87.5%	87.5
Reagent Heel	56.5	4.3%	2.4
Total Input			89.9
SOLIDS OUTPUT			
1 Solids	8	100.0%	8
2 Solids	6.2	100.0%	6
3 Solids	6.3	100.0%	6
Treated Solids	64.2	100.0%	64
Total Output			85
TOTAL INPUT	90		
TOTAL OUTPUT	85		
PERCENT RECOVERY	94.2%		

Test D Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	10	87.5%	8.8
Reagent Heel	27	1.2%	0.3
Total Input			9.1
SOLIDS OUTPUT			
Treated Solids	8.4	100.0%	8
Total Output			8
TOTAL INPUT	9		
TOTAL OUTPUT	8		
PERCENT RECOVERY	92.7%		

Test V1 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	50	33.4%	16.7
Reagent Heel	15.5	1.2%	0.2
Total Input			16.9
SOLIDS OUTPUT			
3 Solids	0.5	100.0%	1
4 Solids	1.3	100.0%	1
Treated Solids	8.8	100.0%	9
Total Output			11
TOTAL INPUT	17		
TOTAL OUTPUT	11		
PERCENT RECOVERY	62.8%		

Test V2 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	50	33.4%	16.7
Reagent Heel	27.1	4.3%	1.2
Total Input			17.9
SOLIDS OUTPUT			
3 Solids	1.2	100.0%	1
4 Solids	2	100.0%	2
Treated Solids	15.5	100.0%	16
Total Output			19
TOTAL INPUT	18		
TOTAL OUTPUT	19		
PERCENT RECOVERY	104.6%		

Test V7 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	50	33.4%	16.7
Total Input			16.7
SOLIDS OUTPUT			
1 Solids	1.6	100.0%	2
Treated Solids	12.1	100.0%	12
Total Output			14

TOTAL INPUT	17
TOTAL OUTPUT	14
PERCENT RECOVERY	82.0%

PHASE II

Soil No.1, Citrate/Dithionite Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	200	92.0%	184
Total Input			184
SOLIDS OUTPUT			
6 Solids	7	100.0%	7
9 Solids	8.7	100.0%	9
Extract Sol'n TSS	9446.7	0.1%	13
Extract Sol'n TSS	9640.1	0.1%	13
Treated Solids	159.2	100.0%	159
Total Output			200

TOTAL INPUT	184
TOTAL OUTPUT	200
PERCENT RECOVERY	109.0%

Soil No.1, Citric Acid/Peroxide Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	200	92.0%	184
Total Input			184
SOLIDS OUTPUT			
6 Solids	9	100.0%	9
9 Solids	10.8	100.0%	11
Extract Sol'n TSS	9081.3	0.0%	2
Extract Sol'n TSS	9242.8	0.0%	3
Treated Solids	144.4	100.0%	144
Total Output			169

TOTAL INPUT	184
TOTAL OUTPUT	169
PERCENT RECOVERY	91.8%

Soil No.2, Citrate/Dithionite Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	200	89.0%	178
Total Input			178
SOLIDS OUTPUT			
6 Solids	6.6	100.0%	7
9 Solids	10.3	100.0%	10
Extract Sol'n TSS	9459.7	0.1%	14
Extract Sol'n TSS	9585.8	0.2%	19
Treated Solids	148.1	100.0%	148
Total Output			197

TOTAL INPUT	178
TOTAL OUTPUT	197
PERCENT RECOVERY	110.7%

Soil No.2, Citric Acid/Peroxide Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	200	89.0%	178
Total Input			178
SOLIDS OUTPUT			
6 Solids	5.5	100.0%	6
9 Solids	7.3	100.0%	7
Extract Sol'n TSS	9671.5	0.1%	7
Extract Sol'n TSS	9652.4	0.0%	4
Treated Solids	133.3	100.0%	133
Total Output			157

TOTAL INPUT	178
TOTAL OUTPUT	157
PERCENT RECOVERY	88.4%

Vegetation, Citrate/Dithionite Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	29.0%	29.0
Reagent	78.5	4.6%	3.6
Total Input			32.6
SOLIDS OUTPUT			
6 Solids	1.5	100.0%	1.5
9 Solids	2.2	100.0%	2.2
Extract Sol'n TSS	4876.8	0.2%	8.6
Extract Sol'n TSS	4891.2	0.1%	6.4
Treated Solids	25	100.0%	25.0
Total Output			43.7
TOTAL INPUT	32.6		
TOTAL OUTPUT	44		
PERCENT RECOVERY	134.0%		